PB84-189851

Controlled Air Incineration of Pentachlorophenol-Treated Wood

Los Alamos National Lab., NM

Prepared for

Industrial Environmental Research Lab.
Cincinnati, OH

May 84

U.S. Department of Commerce
National Technical Information Service
NTIS

PB84-189851

EPA-600/2-84-089
May 1984

CONTROLLED AIR INCINERATION OF PENTACHLOROPHENOL- TREATED WOOD

by

L. A. Stretz and J. S. Vavruska
Waste Management Group H-7
Los Alamos National Laboratory
P. O. Box 1663, MS E517
Los Alamos, New Mexico 87545

USEPA Interagency Agreement
AD-89-F-1-539-0

Project Officer
Richard A. Carnes
Industrial Pollution Control Division
Industrial Environmental Research Laboratory

This study was conducted in cooperation with and with partial support from
Defense Property Disposal Service
Defense Logistics Agency
Federal Center
Battle Creek, Michigan 49061

US Environmental Protection Agency
Industrial Environmental Research Laboratory
Incineration Research Branch
Cincinnati, Ohio 45268

REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. RECIPIENT'S ACCESSION NO.
EPA-600/2-84-089		PB84 189851

4. TITLE AND SUBTITLE	5. REPORT DATE
Controlled Air Incineration of Pentachloro-Phenol-Treated Wood	May 1984
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S)	8. PERFORMING ORGANIZATION REPORT NO.
L. A. Stretz and J. S. Vavruska	
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT NO.
Los Alamos National Laboratory P.O. Box 163, MS E517 Los Alamos, New Mexico 87545	D 109
	11. CONTRACT/GRANT NO.
	IAG AD-89-F-1-539-0
12. SPONSORING AGENCY NAME AND ADDRESS	13. TYPE OF REPORT AND PERIOD COVERED
Industrial Environmental Research Laboratory--Cinn, OH Office of Research and Development U.S. Environmental Protection Agency Cincinnati, OH 45268	Research
	14. SPONSORING AGENCY CODE
	EPA/600/12

15. SUPPLEMENTARY NOTES

16. ABSTRACT

This research was initiated to determine the operating conditions necessary to effect complete thermal destruction (greater than 99.99%) of pentachlorophenol (PCP)-treated wood in a controlled air incinerator (CAI) and to provide a basis for evaluating the applicability of other incineration systems to the destruction of PCP-treated wood. The treated wood in question was scrap from used ammunition crates in Korea. It has been proposed that a substantial amount of such wood be disposed of by incineration in a unit located in that country. A major concern in such incineration is the potential formation of such toxic compounds as chlorinated dibenzo-p-dioxins and dibenzofurans.

Test results showed a combustion efficiency of >99.9% and a destruction efficiency of >99.99% for PCP in the primary chamber under test conditions with no detectable production of tetrachlorodibenzo-p-dioxin (TCDD) or tetrachlorodibenzofuran (TCDF) at detection limits in sample extracts of 1 and 5 ppb, respectively.

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Pentachlorophenol Controlled Air Incineration Destruction and Removal Efficiency	PCP Dibenzodioxins Dibenzofurans GC/MS	Chemistry Engineering Environment

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
		110
	20. SECURITY CLASS *(This page)*	22. PRICE

EPA Form 2220-1 (9-73)

NOTICE

This document has been reviewed in accordance with U.S. Environmental Protection Agency policy and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report describes the results of the experimental evaluation of the thermal destruction of pentachlorophenol (PCP)-treated wood in a controlled air incinerator at the Los Alamos National Laboratory. The disposal of wood treated with PCP has become a significant problem for the US Department of Defense, particularly in Korea, where the sale or burial of treated-wood ammunition crates is prohibited. The purpose of this study was to provide the Department of Defense with information on the destruction efficiency of PCP as a component on wood as a function of various conditions of incinerator operation. This information is to be used to enable an evaluation of whether incinerators in Korea could be used to destroy this material.

This report will also be of interest to those concerned with the incineration of PCP materials in general. Further information on hazardous material research may be obtained through the Incineration Research Branch of the Energy Pollution Control Division of IERL-Ci.

David G. Stephan
Director
Industrial Environmental Research Laboratory
Cincinnati

ABSTRACT

This research was initiated to determine the operating conditions necessary to effect complete thermal destruction (greater than 99.99%) of pentachlorophenol (PCP)-treated wood in a controlled air incinerator (CAI) and to provide a basis for evaluating the applicability of other incineration systems to the destruction of PCP-treated wood. The treated wood in question was scrap from used ammunition crates in Korea. It has been proposed that a substantial amount of such wood be disposed of by incineration in a unit located in that country. A major concern in such incineration is the potential formation of such toxic compounds as chlorinated dibenzo-p-dioxins and dibenzofurans.

A production-scale CAI at the Los Alamos National Laboratory was used to evaluate the destruction efficiency for PCP as a component on treated-wood feed material. This incineration system was originally designed for volume reduction of combustible radioactive wastes. Components of the system include a dual chamber CAI, a water spray quench column, a high-energy venturi scrubber, and a packed-column acid gas absorber followed by an offgas condenser, reheater, high-efficiency particulate air filters, and an activated carbon adsorber.

Test results showed a combustion efficiency of >99.9% and a destruction efficiency of >99.99% for PCP in the primary chamber under test conditions with no detectable production of tetrachlorodibenzo-p-dioxin (TCDD) or tetrachlorodibenzofuran (TCDF) at detection limits in sample extracts of 1 and 5 ppb, respectively.

This report was submitted under the terms of US Environmental Protection Agency Interagency Agreement AD-89-F-1-539-0 and covers the period February 1, 1981, to February 1, 1982. The work was performed at Los Alamos under the US Department of Energy Contract W-7405-ENG-36.

CONTENTS

Disclaimer ii
Foreword iii
Abstract iv
Figures vii
Tables vii
Abbreviations and Symbols viii
Acknowledgments x

1. Introduction 1
2. Conclusions 2
3. Recommendations 3
4. Facility Description 4
   - Incineration 6
   - Offgas Cleaning 6
   - Scrub Solution Recycling 8
   - Ash Removal 9
   - Control and Instrumentation 9
   - Auxiliary Equipment 10
   - Incineration System Dimensions 11
   - Safety Analysis and Environmental Assessment 12
5. Sampling and Sampling Locations 13
   - PCP-Treated Wood Samples 15
   - Ash Samples 15
   - Gas Sampling 15
   - Offgas Cleanup Water Samples 17
   - Miscellaneous Samples 17
6. Test Plan 18
   - Procedural Requirements 18
   - Contaminated Wood Supply 18
   - Operating Conditions 18
   - Test Schedule 19
7. Test Run 25
   - Offgas System Control Settings 25
   - Incinerator 25
   - System Startup 25
   - Test Phase 1, Period 1 26
   - Test Phase 1, Period 2 26
   - Interim Between Test Phases 27
   - Phase 2, Period 1 27
   - Phase 2, Period 2 28
   - Phase 2, Period 3 29
   - Phase 2, Period 4 30
   - Test Run Summary 31

8. Procedures and Analytical Results 32
Sample Preparation 32
Analytical Procedures 33
Analytical Results 37
9. Discussion of Results 39

References 40
Appendixes 41

A. Quality Assurance 41
B. Combustion, Residence Time, and Sample Requirements Calculations 43
C. Destruction Efficiency Calculations 50
D. Field Data Summary 52
E. Combustion Efficiency and Offgas Composition Data 65
F. Sample and Flow Calculations Summary 75
G. Report on Sample Analysis from Southwest Research Institute 77

FIGURES

Number		Page
1	Simplified line drawing of controlled air incineration process	4
2	Offgas cleaning subsystem	5
3	Basic controlled air incinerator	5
4	Scrub solution recycling subsystem	8
5	Building ventilation zoned for containment	11
6	Incineration sample points	13
7	Offgas system sample points	14
8	Hot-zone traverse points (primary chamber outlet)	16
9	Hot crossover duct traverse points (afterburner outlet)	17
10	Average operating conditions during test interval abc	31

TABLES

Number		Page
1	Conceptual Test Schedule: PCP-Treated Wood Run 1	20
2	Planned Operating Conditions	21
3	PCP Run Chronology	22
4	Analysis of PCP Test Samples	38

LIST OF ABBREVIATIONS AND SYMBOLS

ACFM	— actual cubic feet per minute
avg	— average
BSA	— N, O-Bis-(trimethylsilyl)-acrylamide
Btu	— British thermal unit
C	— centigrade
CAI	— controlled air incinerator
CE	— combustion efficiency
DE	— destruction efficiency
DOD	— Department of Defense
DOE	— Department of Energy
DOP	— dioctylphthalate
DPDS	— Defense Property Disposal Service
DRE	— destruction and removal efficiency
ECD	— electron capture detection
EPA	— Environmental Protection Agency
F	— Fahrenheit
FSAR	— Final Safety Analysis Report
ft	— foot, feet
ft^2	— square feet
ft^3	— cubic feet
GADOS	— gravity ash dropout system
GC	— gas chromatography
h	— hour, hours
HEPA	— high-efficiency particulate air
HPLC	— high-performance liquid chromatography
HZ	— hot zone
IAG	— interagency agreement
i.d.	— inside diameter
ID	— induced draft
in.	— inch, inches
in. Hg	— inches of mercury (pressure)
kg	— kilograms
kJ	— kilojoules
kPa	— kilopascals
L	— liters
lb	— pounds
m	— meters
m^3	— cubic meters
max	— maximum
m/e	— mass to charge ratio
min.	— minimum
min	— minutes
mL	— milliliters

mm	—	millimeters
mol	—	moles
MS	—	mass spectrometry
ng	—	nanograms
OG	—	offgas
PCP	—	pentachlorophenol
PIC	—	product of incomplete combustion
POHC	—	primary organic hazardous constituent
ppb	—	parts per billion
P-P-I	—	phase - period - interval
ppm	—	parts per million
psi	—	pounds per square inch
psia	—	pounds per square inch absolute
QA	—	quality assurance
R	—	Rankine
s	—	seconds
SCF	—	standard cubic feet
SCFM	—	standard cubic feet per minute
SIM	—	selected ion monitor
SWRI	—	Southwest Research Institute
T	—	tons
TCDD	—	tetrachlorodibenzo-p-dioxin
TCDF	—	tetrachlorodibenzofuran
TIC	—	total ion current
TMCS	—	trimethylchlorosilane
TRU	—	transuranic
TSIM	—	N-trimethyl-silyl-imidazole
W.G.	—	water gauge
wt	—	weight
ΔP	—	pressure differential
µg	—	micrograms
µL	—	microliters
µm	—	micrometers

ACKNOWLEDGMENTS

This study was performed at the Los Alamos National Laboratory, which is operated for the US Department of Energy by the University of California under contract number W-7405-ENG-36.

The efforts of the staff and technical members of Los Alamos National Laboratory Waste Management Group H-7 in performing the experimental work and the Industrial Hygiene Group H-5 in analytical support are gratefully acknowledged.

SECTION 1

INTRODUCTION

Incineration under inadequate conditions or open burning of wood which has been treated with pentachlorophenol (PCP) can generate toxic products of incomplete combustion (PICs). Various chlorinated organic compounds can occur in combustion products from burning PCP-treated wood, including chlorinated dibenzofurans and chlorinated dibenzodioxins such as 2, 3, 7, 8 tetrachlorodibenzo-p-dioxin (TCDD).[1-4] The controlled air incinerator (CAI) at the Los Alamos National Laboratory is capable of incinerating the treated wood safely while combustion products are studied and required operating conditions and procedures are established to avoid production of secondary hazardous materials.

The safe disposal of wood treated with PCP has become a significant problem for the US Department of Defense (DOD), particularly in Korea. Wooden ammunition crates are treated with PCP to prevent decomposition of wood due to termite or other insect/borer infestation during transportation and storage. Disposal of the empty crates by sale or burial is prohibited in Korea. The volume of wood involved is so large that shipment back to the United States is an expensive and unattractive option. Incineration of the crates in Korea has been proposed as the most cost effective and environmentally acceptable solution.

The Korean government has asked for evidence that incineration of the material will not create an additional, perhaps more severe, problem than the PCP-treated wood itself presents. DOD, through the Defense Property Disposal Service (DPDS), is seeking operating data on PCP-treated wood incineration to demonstrate that an existing incinerator in Korea could safely destruct the material.

In response to discussions with Environmental Protection Agency (EPA) staff and representatives of the DPDS, an experimental evaluation of the thermal destruction of PCP on wood was performed at Los Alamos National Laboratory by Waste Management Group H-7. The purpose of the evaluation was to determine the destruction efficiency for PCP fed as a component on treated wood while simulating conditions obtainable in a potential disposal incinerator in Korea.

SECTION 2

CONCLUSIONS

PCP-treated wood can be incinerated in the Los Alamos CAI unit with a destruction efficiency (DE) greater than 99.99%. Testing showed no evidence of TCDD at a detection limit of 1 ppb or of tetrachlorodibenzofuran (TCDF) at a detection limit of 5 ppb using gas chromatography/electron capture detection. The DE is greater than 99.99% for the primary chamber alone, indicating that such DE can be accomplished in a single-chamber unit if proper conditions are maintained.

Uncontrolled burning which occurred during upset conditions yielded some evidence of unburned hydrocarbons, although these could not be verified as PCP. This result indicates that such conditions could yield low DEs and/or generation of unwanted PICs. The specific conditions were low temperature [<800°C (1480°F)] and insufficient oxygen (substoichiometric). Avoiding these conditions should result in the desired 99.99% DE or >99.9% combustion efficiency (CE).

SECTION 3

RECOMMENDATIONS

Incineration is a disposal method for PCP-treated wood which can achieve >99.9% CE (>99.99% DE) for PCP if operating conditions are maintained above acceptable minimums and the feed rate is low enough to allow complete combustion of the wood. Test results using the Los Alamos CAI system indicate that a single-chamber incinerator should provide >99.9% CE (>99.99% DE) if the following conditions are maintained:

- minimum combustion chamber temperature >980°C (1800°F),
- retention time (gas phase) >2.5 s,
- excess air >20% (oxygen >3% in offgas),
- adequate time between feed cycles for ash burnout, and
- avoidance of overfeeding that could cause low instantaneous oxygen level and smoke formation.

If maintaining the given conditions at the nominal feed rate for a proposed incinerator is not possible, the unit should be adjusted to a feed rate at which the conditions can be met.

Sampling the offgas, scrubber effluent, and ash as part of any disposal operation is also recommended to verify that satisfactory destruction is occurring and that toxic materials are not present in the secondary waste streams. This sampling is essential, at least until successful operation of the subject incinerator is verified, since different incinerator configurations can have different combustion characteristics at "identical" operating conditions.

SECTION 4

FACILITY DESCRIPTION

The Los Alamos CAI is a system which has been developed through careful modification and integration of commercially available equipment. The system was designed and constructed to demonstrate technology for the combustion of transuranic (TRU) contaminated waste (alpha-bearing radioactive waste). As a result, the system is equipped with sophisticated containment, safety, and offgas cleanup systems which make it suited to hazardous material incineration studies.

The basis of the Los Alamos CAI is an Environmental Control Products (Charlotte, North Carolina) model 500-T incinerator. Many operational modifications made at Los Alamos are now available as standard options on the basic unit. Such modifications include fully modulated burners, gravity ash removal, steam injection capability, and improved mixing of secondary air with primary chamber effluent. Many other modifications to the standard unit have been made. The basic incinerator and associated offgas cleaning equipment are available on the commercial market.

The more prominent features of the Los Alamos CAI system are shown in simplified line drawings (Figures 1-3) and a discussion of the incineration system is given in this section. A more detailed facility and process description is given in the facility Final Safety Analysis Report (FSAR).[5]

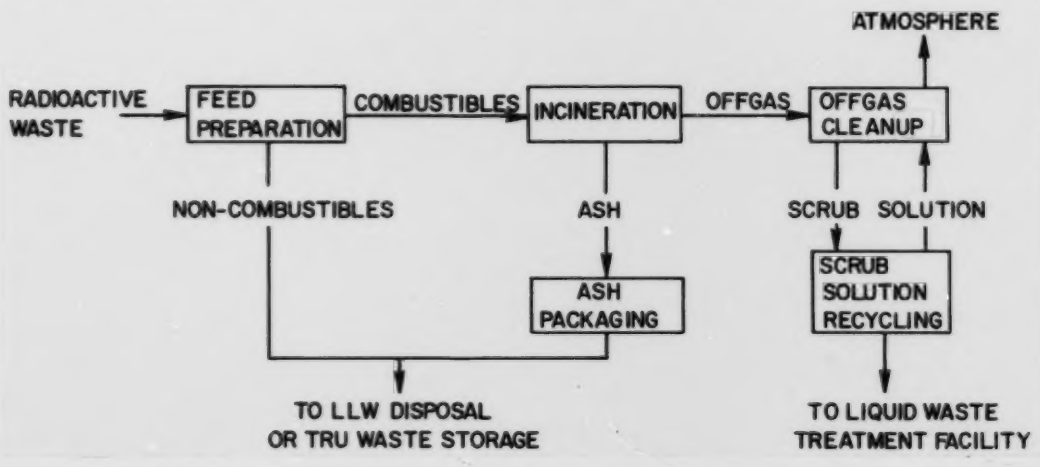


Figure 1. Simplified line drawing of controlled air incineration process.

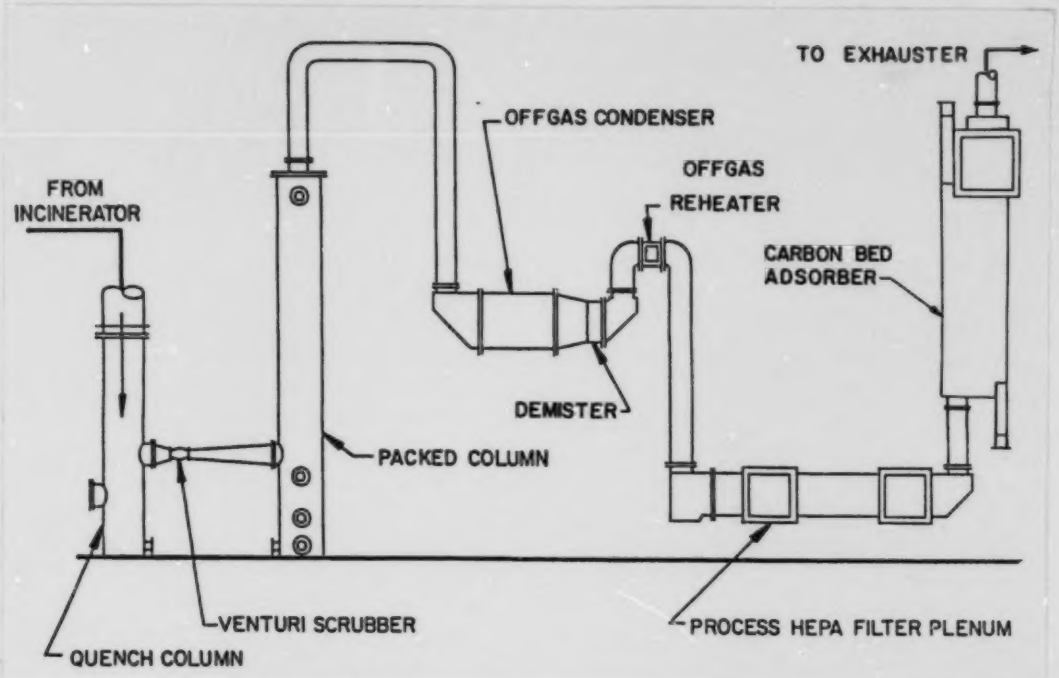


Figure 2. Offgas cleaning subsystem.

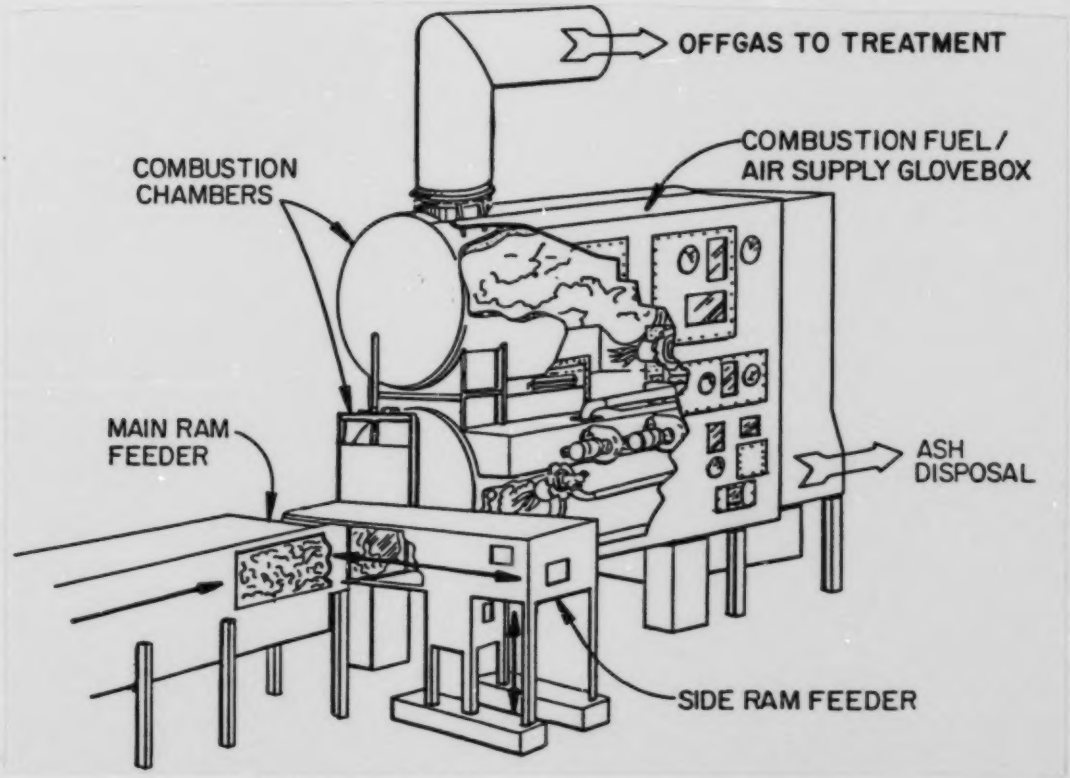


Figure 3. Basic controlled air incinerator.

4.1 INCINERATION

The incinerator is a conventional design, dual chamber, controlled air, solid waste system which has been modified to provide physical containment barriers at all pathways between the incinerator interior and the process area. These modifications have been made to enhance safety and containment when incinerating radioactive materials and also to increase the level of protection for operators and the surrounding area when incinerating other hazardous materials.

The standard unit has a nominal capacity of 170 kg (374 lb)/h of Type-O waste at sea level and is also rated for waste Types 1-4 and Public Health Service type waste. The Los Alamos unit has been derated due to the lower atmospheric pressure [2225-m (7300-ft)] elevation of the site and has been demonstrated at 45.4 kg (100 lb)/h of a waste containing a mixture of 35% cellulosics, 23% polyethylene, 12% polyvinylchloride, and 30% rubber (primarily polyisoprene, latex, and neoprene) and having a heating value of 30.5 kJ/g.

The primary chamber is a refractory-lined cylinder with an inside diameter of 1.46 m (4.8 ft) and a length of 1.83 m (6 ft). The secondary chamber is 1.17-m (3.8-ft) i.d. and 1.83 m (6 ft) long. The crossover duct to the offgas cleanup system is also refractory lined and maintains the offgas at near the secondary chamber temperature, thus acting as an extension of the chamber. This duct has an inside diameter of 0.51 m (1.7 ft) and an overall length of 4.14 m (13.6 ft). The resulting effective chamber volumes are 3.06 m^3 (107 ft^3) for the primary chamber and 1.97 m^3 (68 ft^3) for the secondary chamber (afterburner excluding the duct). Burners in the primary and secondary chambers are both fully modulated for temperature control. Maximum operating temperatures are nominally 1370°C (2500°F) in both chambers. Constraints on the glovebox ventilation system limit the temperature achievable to 1230°C (2250°F) in the current configuration.

Incineration temperature is controlled by feedback loops on both combustion chambers. The temperature sensor on the primary chamber is located in the transition duct at the chamber exit. Secondary chamber temperature is measured in the offgas duct just downstream of the chamber outlet. Additional thermocouples are located at the refractory surface in both chambers and at the end of the hot offgas duct upstream of the quench column.

Operation of the unit with solid waste is a quasi-continuous process with waste charged through the main ram controlled by an interval timer. The timer is set as a function of desired feed rate and waste density. For example, waste packaged in 0.06-m^3 (2-ft^3) boxes with a density of 160 kg (352 lb)/m^3 to be fed at a nominal rate of 45.4 kg (100 lb)/h would be fed at 12-min intervals with the ram automatically activated by the cycle timer. Ash can also be removed on-line by manual activation of the gravity ash dropout system (GADOS). This is normally done only when the ash pile becomes excessive.

Underfire and secondary combustion air rates are independently controlled and can be adjusted to give the desired O_2 concentration in the gas effluent stream.

4.2 OFFGAS CLEANING

Exhaust from the CAI upper chamber contains both particles and mineral acids which result from combustion of the feed material. These chemical pollutants and particles are removed by

the offgas cleaning system, which consists of a quench tower, a high-energy venturi scrubber, a packed-column absorption tower, a condenser, a mist eliminator, a reheater, high-efficiency particulate air (HEPA) filters, an activated carbon adsorber, and an induced-draft (ID) blower (Figure 2).

The quench tower is divided into an upper contacting section and a lower separating section. Combustion gases are cooled from the incinerator exit temperature to approximately 70°C (160°F) by evaporation of recycled scrub solution. Excess solution collects in the separator while the saturated gas phase is routed to the inlet of the venturi scrubber.

The variable-throat venturi scrubber, located between the quench tower and the absorption tower, provides high-efficiency removal of the offgas particles. The venturi assembly consists of converging and diverging cones with a clamp valve throat to allow the pressure drop to be controlled. Venturi pressure drop is normally controlled to 14.9 kPa (60 in. W.G.). Scrub solution is injected through a nozzle located upstream of the throat.

Residual mineral acids are removed from the gas phase by countercurrent contact with process condensate, recycled scrub solution, or makeup water in the packed-column absorption tower.

The condenser, mist eliminator, and reheater are included to condition the process exhaust gases before final HEPA filtration. The condenser lowers the offgas temperature to approximately 45°C (110°F), removing most of the water vapor from the scrubbed gas stream. The offgas is then reheated approximately 17°C (30°F) above the saturation temperature to about 62°C (144°F), to avoid condensation and attendant plugging of the HEPA filters and corrosion of the plenum, carbon adsorber, exit ducting, and offgas blowers. The functional parts of each of these subsystem components are commercial equipment, housed in enclosures specially designed to withstand the 24.8-kPa (100-in. W.G.) pressure differential between the process and ambient conditions.

HEPA filtration is required for final removal of particles in radioactive service. The HEPA filters remain in use for hazardous waste testing. The filter module houses two frames in series, the first consisting of a prefilter and two HEPA filters, the second being similar but without the prefilter. The filter housing is designed to withstand the 29.8-kPa (120-in. W.G.) pressure differential capability of the process and is fitted with hatches to access the bagout doors and in-place filter testing ports. The filters are tested with dioctylphthalate (DOP), and performance is better than 99.97% removal of 0.3-μm particles.

An activated carbon adsorber on the full offgas flow is located downstream of the HEPA filters. This adsorber provides additional protection against release of organics through the process stack. The bed provides a 0.5-s residence time at a superficial velocity of 0.25 m/s (50 ft/min) for maximum offgas flow and approximately 2 s at normal offgas flow rates.

The ID blower is capable of producing 57.2-kPa (230-in. W.G.) static pressure absolute at 53.8 m^3 (188 ft^3)/min with a discharge pressure of 78.0 kPa (314 in. W.G.) absolute to accommodate the 2225-m (7300-ft) elevation at Los Alamos.

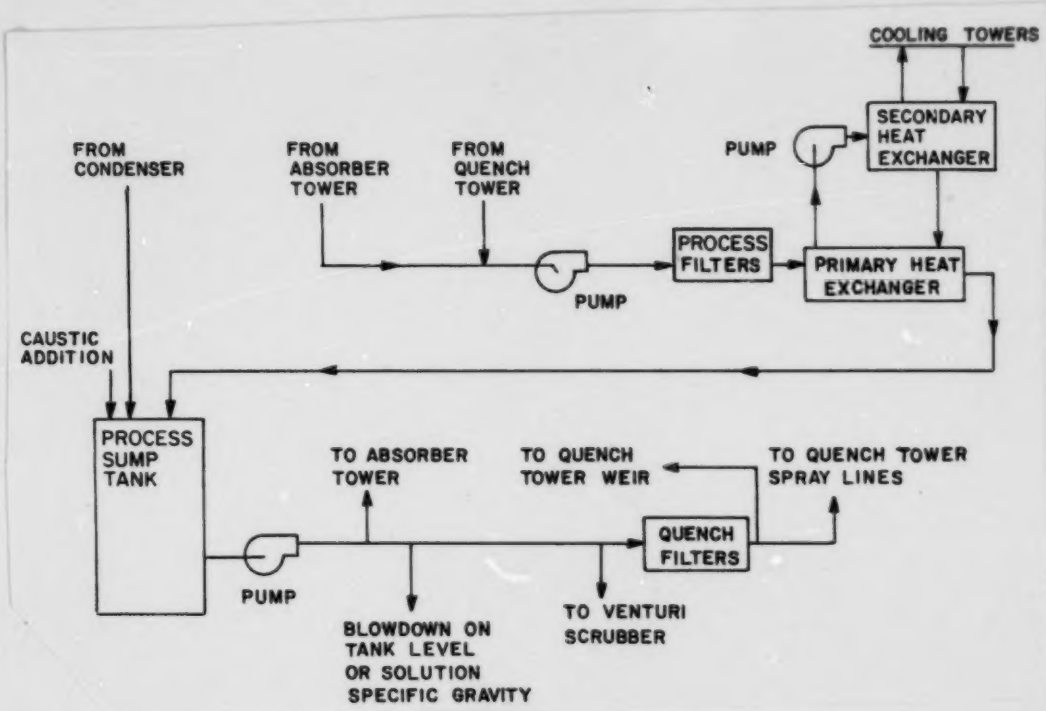


Figure 4. Scrub solution recycling subsystem.

4.3 SCRUB SOLUTION RECYCLING

A scrub solution recycle system is used to minimize liquid blowdown (waste to liquid treatment) from the offgas cleaning system (Figure 4). This system consists of full-flow cartridge liquid filters, a graphite heat exchanger, two evaporative cooling towers, a scrub solution receiver tank, a condensate receiver tank, and a caustic (20% NaOH) makeup tank. Quench tower liquid effluent combines with scrub solution and venturi blowdown from the base of the packed-column absorber tower. This solution is pumped through 100-μm cartridge filters and a primary heat exchanger to the receiver tank. Liquid requirements for the quench tower, venturi scrubber, and absorption tower are satisfied by recycle from the receiver tank. Solution recycling through the quench tower is filtered to remove particles down to 20 μm.

The graphite heat exchanger cools recycling solution from 85°C to 50°C (185°F to 120°F). The process (tube) side is operated at a lower pressure than the coolant (shell) side to guarantee in-leakage in the event of tube failure. The shell side fluid from the primary heat exchanger is cooled by the secondary heat exchange loop, providing isolation from the environment.

To control scrub solution acidity, 20% caustic (NaOH) solution is added at the receiver tank inlet. The addition rate is controlled by a pH sensor on the outlet of the receiver tank, and pH is controlled within the range of 8-9. Condensate from the condenser/mist eliminator drains into a condensate receiver tank. The level in this tank is maintained by addition of fresh water. The solution is then pumped either to the top of the packed-column scrubber or to the receiver tank.

The blowdown rate from the scrub solution receiver tank is controlled by liquid level and specific gravity. If the specific gravity of the scrub solution exceeds a specified value (currently 1.05) or if the liquid level becomes excessive, the rate of blowdown which is sent to the liquid waste treatment plant is increased.

4.4 ASH REMOVAL

Ash removal from the CAI is accomplished through one of two paths. A GADOS is used for ash removal during operation and a vacuum ash removal system is used for thorough cleanout of both chambers of the incinerator during shutdowns.

The GADOS consists of a refractory-lined pit and door in the floor of the primary chamber of the CAI located at the end of the hearth opposite the ram feeder. As new waste is fed to the incinerator, the ash is pushed down the hearth until it drops into the ash removal pit. Periodically, the dropout door is opened for a brief time to allow the ash to fall through a grate and delumper wheel into a collection hopper. The ash is then vacuumed from the GADOS hopper and collected by a high-energy cyclone and sintered metal filter system into a second hopper for removal at the ash packaging station.

The vacuum system, which is capable of producing up to 20.8-kPa (84-in. W.G.) suction, is also used for vacuum ash cleanout during shutdowns. This cleanout is achieved by manipulating a vacuum hose in the incinerator chambers through the access doors and gloveboxes on the ends of the chambers.

The ash packaging station consists of a glovebox where ash is removed from the collection hopper through an interlocked isolation chamber. The chamber is first opened to the ash hopper and allowed to fill, then isolated from the hopper and opened to drop the ash into a collection bag.

The ash is then packaged and stored for future studies or immobilization.

4.5 CONTROL AND INSTRUMENTATION

Design and specification of the CAI process control system received priority throughout the planning and construction phase. As the "nerve center" for the process, the controls not only assure effective component performance but also monitor performance data to assure the safety of continuing operations. Control design considerations started with the parameters affecting operation of the incinerator and primary offgas components. As the process design evolved, considerations broadened to encompass the operation of ancillary equipment including back-up utility supplies. A detailed description of the complete control system is well beyond the scope of this document; however, the incinerator control features and a general overview of the total system design are described below.

Controls on the as-received incinerator were largely limited to pre-set conditions based on combustion experience for a particular waste composition. These controls were upgraded to accept a wider range of feed compositions and to minimize thermal cycling in the lower chamber. Both air and natural gas supplies are modulated to permit proportional rather than step response to demand. The air introduction rate for each stage can be controlled by

feedback from oxygen analyzers located at the exit of each chamber. The pressure differential between the incinerator interior and the operating area is maintained by a valve immediately upstream of the ID fan. Flow measuring elements and recorders monitor air, natural gas, and steam introduction rates for energy and material balance purposes.

For offgas cleanup, conditioning, and filtration equipment downstream of the incinerator, the controlled variables are (1) venturi scrubber liquid feed rate and pressure drop; (2) absorber tower liquid feed rate; (3) condenser gas-phase temperature decrease; and (4) reheater gas-phase temperature increase. The pressure drop and nominal temperature of each component are also monitored as an indication of normal versus deteriorating performance.

In the scrub solution recycle subsystem, a pH feedback arrangement controls neutralization of the liquid effluent from the primary offgas scrubbing components. Differential pressure is monitored across each liquid filter, while process side temperatures are controlled in the graphite heat exchanger. Liquid level and specific gravity are controlled in the scrub solution receiver tank.

Primary variables, and many secondary variables and parameters, are controlled and/or recorded at a central station. Variables considered critical to process operation and safety are tied into an alarm panel which positively identifies the off-range variable as an aid to troubleshooting. Off-range variables identified as vital to process safety will activate one of three automatic shutdown modes—controlled, fast, or emergency. Less critical alarmed variables require only operator response to correct off-range behavior.

A data acquisition system automatically records the many variable and parameter values generated during experimental CAI process runs.

A more detailed description of the safety and automatic shutdown systems is given in the FSAR.[5]

4.6 AUXILIARY EQUIPMENT

Backup utilities provide required services for an orderly process shutdown under abnormal circumstances. A diesel-powered generator with automatic switchgear is kept running during all incinerator operations. The unit supplies standby power to high-consumption equipment and vital motor-driven equipment such as the ID fans to permit safe operation and shutdown in case of a main power failure. An on-line, floating battery system provides electrical power for process controls and data collection, averting potential momentary power interruptions which could result in control relay dropout. A 2-h auxiliary cooling water supply is stored in a pressurized container for release to the quenching system in the event of a recirculation pump failure which otherwise would present a threat of damage to process equipment. A back-up air compressor and compressed nitrogen are available to supplement normal instrument air supply if required. Pneumatic actuators are designed to "fail safe" on loss of air pressure. Snuffing steam can be injected into the primary chamber to extinguish burning waste in the event of a fast shutdown at high temperature. This capability prevents uncontrolled burning and inefficient combustion, which can clog the offgas cleaning system with soot and heavy tars and lead to release of toxic contaminants.

Containment for the building is maintained by physical barriers and by four separate ventilation zones. The pressure of each zone is regulated so that ventilation air moves from the highest pressure zone (atmospheric) toward the lowest pressure zone (the volume internal to the process). The interface between each zone is controlled by physical enclosures. The zone ventilation system is shown in Figure 5.

4.7 INCINERATION SYSTEM DIMENSIONS

Dimensions of the incineration system components through the absorption column are

primary chamber	1.46-m i.d. × 1.83 m long (4.8 ft × 6 ft),
transition duct	0.31-m × 0.91-m i.d. × 0.61 m long (1 ft × 3 ft × 2 ft),
secondary chamber	1.17-m i.d. × 1.83 m long (3.8 ft × 6 ft),
hot offgas duct	0.51-m i.d. × 4.14 m long (1.7 ft × 13.6 ft),
quench column	0.61-m i.d. × 1.83-m contact length (2 ft × 6 ft),
venturi scrubber	variable throat × 1.22-m (4-ft) overall length, and
absorption column	0.61-m i.d. × 3.05-m (2-ft × 10-ft) contact length.

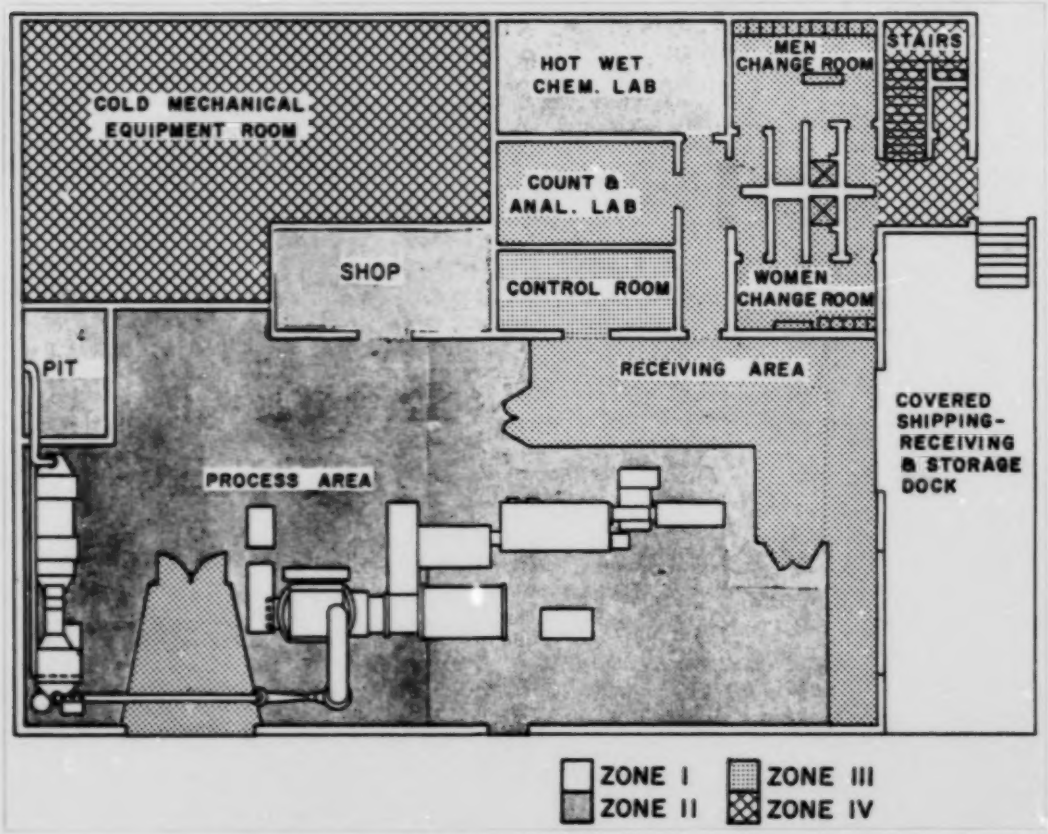


Figure 5. Building ventilation zoned for containment.

4.8 SAFETY ANALYSIS AND ENVIRONMENTAL ASSESSMENT

The facility housing the Los Alamos CAI has been subjected to a detailed safety analysis,[8] including environmental assessment for operation with plutonium-bearing waste. Though many hazardous waste concerns are similar to those for radioactive waste, the FSAR has been reviewed to assure that the work force, public, and environment are adequately protected.

SECTION 5

SAMPLING AND SAMPLING LOCATIONS

Sampling and analysis of all streams are essential during testing to evaluate incinerator performance, to evaluate offgas system performance, and to detect and quantify products of incomplete combustion or chemical reactions. Figures 6 and 7 show the location of sampling points and data collection related to process conditions. Samples of the wood material were subjected to PCP analysis to determine the PCP content in the incinerator feed material. Appendix A discusses quality assurance (QA) measures taken to validate the testing.

The configuration of the incinerator and offgas system does not allow isokinetic sampling upstream of the quench and scrub system. Nonisokinetic samples were taken from the hot-zone interchamber and from the hot flue-gas duct upstream of the quench column. Sampling trains on the hot-zone and hot flue-gas sample points were modified EPA-Method 5 trains, including a column of solid sorbent polymer (XAD-2®). Each sample train was used to collect samples for a 4-h period based on preliminary estimates of detection limits, conversions, and feed concentrations. A second 4-h sample was collected at each point before modifying the combustion conditions. The preliminary calculations which led to selection of a 4-h sample

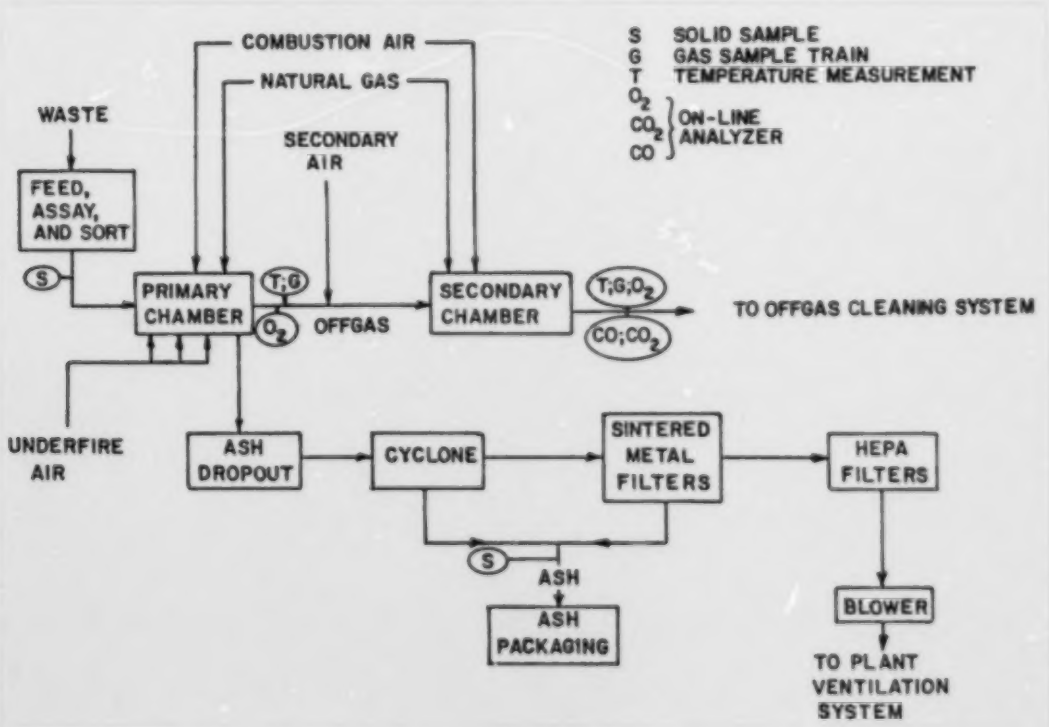


Figure 6. Incineration sample points.

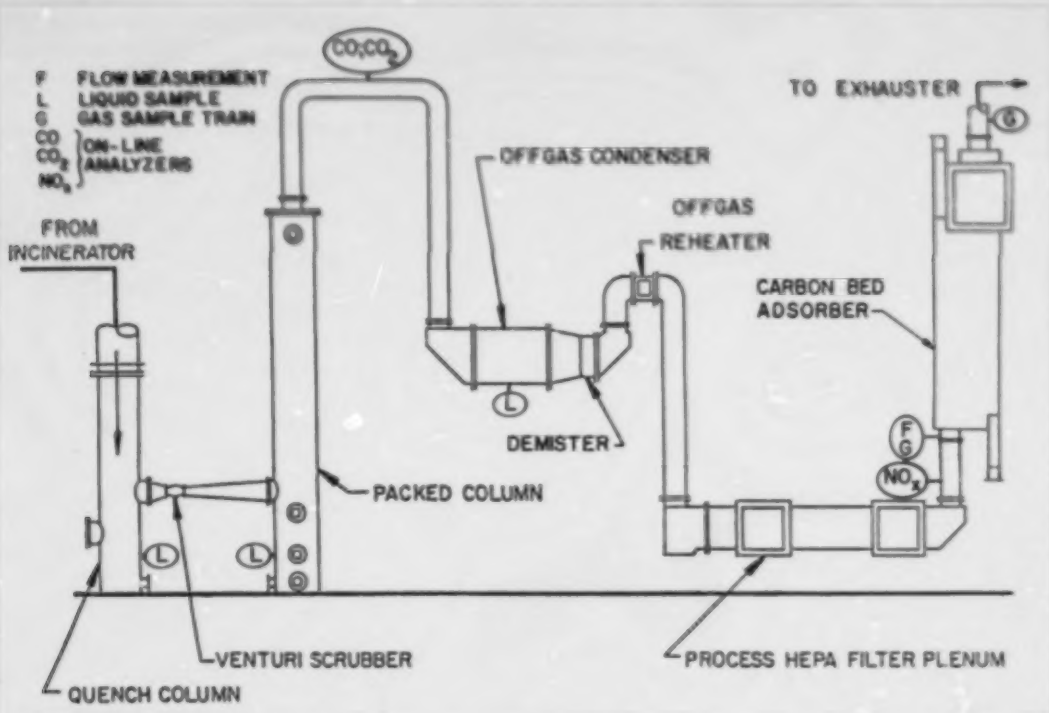


Figure 7. Offgas system sample points.

period are shown in Appendix B along with recalculations based on actual run data. Gas samples were also taken downstream of the HEPA filters and downstream of the carbon bed adsorber prior to discharge through the facility stack. These samples were passed through a XAD-2® column for organics collection, and the final sample was passed through NaOH solution for HCl determination.

Solid samples of the feed material and bottom ash were taken to provide data on the quantity of PCP fed to the incinerator that remained uncombusted in the ash. The feed sample was used to identify the principal organic hazardous constituents (POHCs) as required in the EPA incinerator standards published in the January 23, 1981, Federal Register.[8] The bottom ash samples were collected at the ash packaging station following discharge from the incinerator and conveying to the collection system. Ash samples were collected after each transfer and retained in separate containers.

Post-run samples were taken of the scrub liquid filters. No material was present on the HEPA filters.

In addition to the usual incinerator control instrumentation, on-stream instrumentation included combustibles/O_2 analysis on the incinerator interchamber and on the secondary chamber discharge, CO and CO_2 analysis at the secondary chamber discharge and the absorber tower discharge, and final flow measurement on the HEPA filter plenum discharge. The combustibles/O_2 analyzer is used primarily for O_2 analysis. As a safety feature, the instrument also indicates when excess fuel (as total hydrocarbons) is present. Auxiliary fuel gas flow is controlled on temperature, and burner combustion air flows are controlled on ratio to

fuel gas. Auxiliary fuel gas, burner air, underfire air, secondary air, and final offgas flows are all measured and recorded.

Samples were analyzed at Los Alamos facilities and at Southwest Research Institute (SWRI), San Antonio, Texas. Gas analyses were done with gas chromatography (GC) and/or systems with various detectors including a GC-mass spectrometer system (MS) with a computerized data base for species identification and quantification. More details on analytical procedures are provided in Sec. 8. Detailed sampling information is given in the following subsections.

5.1 PCP-TREATED WOOD SAMPLES

Samples of PCP-treated wood were collected at random from the more than 2 T of smashed ammunition crates supplied by DPDS for the test burn. The wood samples were Soxhlet extracted with toluene at 102°C for 24 h and the extract analyzed as detailed in Sec. 8 to determine the PCP content of the feed material. These samples determined POHCs in the feed material as required by the EPA incinerator standards published in the Federal Register.[8] Chlorine content of the feed material was found to be less than 0.08 wt%.

5.2 ASH SAMPLES

Samples of ash were collected during ash transfer from the incinerator and held in separate containers. The incinerator primary chamber was vacuumed between the untreated wood and PCP-treated wood phases of the test to assure that the treated-wood ash was not diluted.

A portion of each ash sample was Soxhlet extracted with toluene at 102°C and the extract subjected to analysis as detailed in Sec. 8.

5.3 GAS SAMPLING

Four locations were selected for gas/particulate sampling in the incineration and offgas systems. System configuration and radioactive contamination from previous testing limited sampling capabilities. However, adequate sampling was achieved by following the procedures detailed below for each sample location.

5.3.1 Hot-Zone Sample

Hot-zone samples were collected from the connecting duct between the primary and secondary incineration chambers. The duct is rectangular in cross section with dimensions as shown in Figure 8. The sampling train used at this location was a modified EPA-Method 5 train with a high-temperature probe. The probe was stainless steel with water jacket cooling. The filter holder and filter were omitted from this train and the particulate collected in the cyclone separator and impingers. The first impinger contained 250 mL of toluene and was followed by two impingers containing 250 mL each iso-octane (2, 2, 4 trimethyl pentane). A column containing solid XAD-2® sorbent was included between the third and fourth impingers to collect any trace organics not captured in the solvent impingers. The fourth impinger contained 200 g

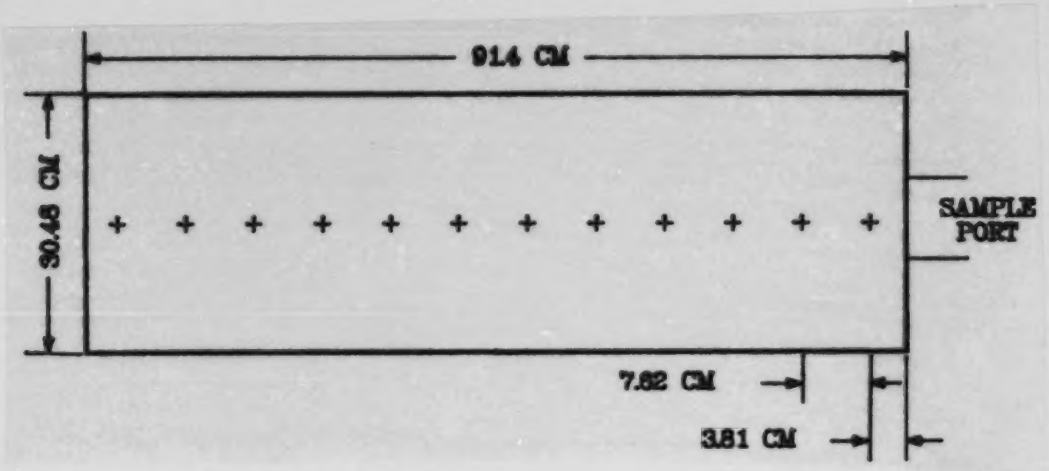


Figure 8. Hot-zone traverse points (primary chamber outlet).

(7 oz) of indicating silica gel to collect moisture from the gas sample upstream of the pump, gauges, and dry gas meter. All impingers were cooled by immersion in an ice bath.

The sampling probe was limited to a single sample plane as shown in Figure 8, and each 4-h sample included 20 min at each of 12 traverse points in the duct.

5.3.2 Hot Offgas Sample

The hot offgas samples were collected from the offgas duct upstream of the quench column. The duct dimensions and sample location are shown in Figure 9.

The sampling train was similar to the train described in Sec. 5.3.1 except that the probe was stainless steel with a tantalum nozzle. The probe was limited to a single plane, and samples were taken at equal intervals during each 4-h sample period at the 10 traverse points shown in Fig. 9. Nominal sample flow was 14.2 L (3.7 gal)/min at standard conditions.

5.3.3 Downstream Gas Samples

Two sample locations were downstream of the incinerator offgas cleanup system. The offgas system includes not only the quench high-energy scrubber absorber system but three stages of HEPA filters and an activated carbon adsorber. The HEPA filters, which are nuclear grade, have been tested with dioctylphthalate to pass a minimum efficiency criterion of 99.97% of 0.3-μm particles.

The first downstream sample point followed two banks of these filters and the second followed the full-flow carbon bed adsorber and a third bank of HEPA filters. These samples were of a homogeneous gas phase with essentially no particles. Therefore, the sample trains at these locations did not require a sample probe and traverse. Samples were withdrawn through a tube open to the gas stream. The collection train at both locations consisted of a single impinger containing 250 mL of iso-octane followed by a XAD-2® trap and sample monitoring, controlling, and pumping equipment. Desired sample flow was 14.2 L (3.7 gal)/min at standard conditions.

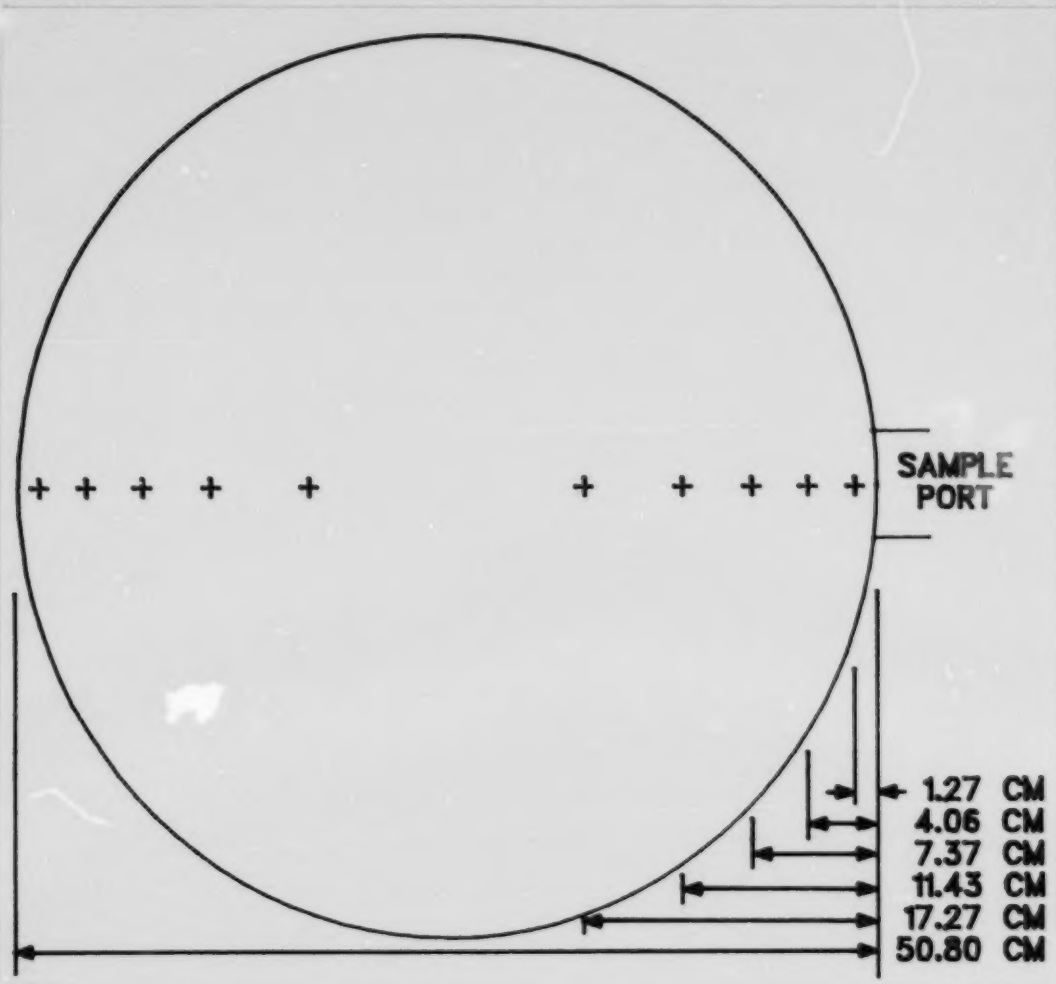


Figure 9. Hot crossover duct traverse points (afterburner outlet).

5.4 OFFGAS CLEANUP WATER SAMPLES

Samples of the offgas stream scrub solution were taken during each sample period and were extracted with toluene to capture TCDD or other chlorinated organics from the solution. The organic fraction was separated and saved for analysis as described in Sec. 8.

Sample locations for scrubber liquid included the quench column sump, absorber column sump, and recycle solution. An additional sample was taken of the condensate from the offgas condenser.

5.5 MISCELLANEOUS SAMPLES

Other samples were taken whenever operating events occurred that could potentially remove material of interest from the system. Such events included changing scrub solution filters during the run and inspecting HEPA filters after the run.

SECTION 6

TEST PLAN

The purpose of the testing was to determine the destruction efficiency for PCP-treated wood as a feed component to the Los Alamos CAI simulating conditions obtainable in a proposed disposal incinerator in Korea. Potential existed for studying the effects of feed rate, incinerator temperature, and excess air (and, indirectly, residence time) on the destruction efficiency (DE) for PCP. Operating conditions included those which would best model the incinerator proposed for final disposal of the subject feed material.

Available information on the proposed incinerator indicates that it is a single-chamber, oil-fired, forced-draft unit. Waste is manually fed to the combustion chamber, which operates at variable temperatures up to a maximum of 1200°C (2192°F). These conditions could be modeled best by operating the Los Alamos unit and sampling between the primary and secondary chambers to evaluate the effectiveness of a single chamber. Information on temperature profiles, feed rates, and offgas flow rates for the proposed disposal incinerator was not available; therefore, the testing was designed to evaluate performance at several temperature/residence time values which should be obtainable in the proposed unit.

6.1 PROCEDURAL REQUIREMENTS

Before conducting test burns, several procedural steps were necessary. Since the facilities at Los Alamos are operated for the US Department of Energy (DOE), an Interagency Agreement (IAG) between DOE and EPA was required before testing could begin. This agreement was secured as IAG No. AD-89-F-1-539-0.

6.2 CONTAMINATED WOOD SUPPLY

PCP-treated wood for testing was as similar as possible to the treated wood for which disposal options were being evaluated. Actual ammunition crate wood was supplied for use in the test runs by DPDS, a co-sponsor of this program.

The ram feeder system on the CAI is designed to load 0.31-m x 0.31-m x 0.62-m (1-ft x 1-ft x 2-ft) boxes or bundles of waste. Slightly larger loading configurations are possible, but modification of the system to alter the feed configuration significantly did not appear to be warranted. Therefore, the wood was packaged in the standard boxes at a density of 160.2 kg/m^3 (10 lb/ft^3) or 9.08 kg (20 lb)/box and fed through the existing feed train.

6.3 OPERATING CONDITIONS

The test plan was designed to provide combustion data over a range of conditions to qualify a proposed incinerator for PCP-treated wood disposal. Input from the DPDS and EPA was

requested for use in establishing initial test operating conditions to produce the maximum of useful data. The CAI can be operated at a wide variety of conditions utilizing temperature control on chambers with combustion and secondary air control to establish desired levels of excess air. Operating the system at a variety of conditions seemed desirable to provide parametric data as well as to model the proposed Korean incinerator. The run was separated into two phases: Phase 1 with untreated wood and Phase 2 with PCP-treated wood. The second phase consisted of four test periods at various operating conditions.

6.4 TEST SCHEDULE

The first phase involved system startup and operation with untreated wood to verify that the system was functioning properly with no major problems related to complete combustion of the wood. This phase was also to be used to obtain baseline (blank) samples for analysis. The second phase involved testing with PCP-treated wood at conditions specified to simulate the candidate system and to study the combustion characteristics of the PCP-treated wood. Table 1 gives the planned basic test schedule and Table 2 gives the planned operating conditions (experimental design).

The actual test run was about 1 day longer than planned due to several operational problems during the test period. A run chronology is given in Table 3. Problems which may have impacted sample results and deviations from the experimental design are discussed in Sec. 7.

TABLE 1. CONCEPTUAL TEST SCHEDULE: PCP-TREATED WOOD RUN 1

Test day	Time	Activity
1	0815	Begin startup.
	0915	Fire lower chamber burner.
		Fire upper chamber burner as needed.
		Heatup at 38°C (100°F)/h.
2	0515	Lower chamber at 925°C (1700°F).
	0715	Upper chamber at 1010°C (1850°F)
	0730	Collect liquid samples and blanks from particulate and offgas sample trains.
	0830	Begin untreated wood feed [45.4 kg (100 lb)/h], period 1.
		Begin offgas sample collection.
	1230	Collect samples.
	1800	Begin second sample interval.
	1900	Collect samples.
	1930	Adjust operating conditions.
	2100	Begin phase 1, period 2 sampling.
3	0100	Collect samples.
	0230	Begin second period.
	0630	Collect samples.
	0730	Begin cooldown.
4		Continue cooldown.
5	0200	Open lower chamber to vacuum ash from hearth. Collect ash sample.
	0600	Relight burners, begin heatup for PCP-treated wood phase.
6	1400	Verify operation of all sampling, analysis, offgas cleanup, and safety systems. Unit at 927°C (1700°F) lower, 1204°C (2200°F) upper.
	1430	Start PCP-treated wood feed at 27.2-kg (60-lb)/h rate, operating conditions as given in phase 2, period 1, of Table 2.
	1830	Collect samples.
	1930	Begin second sample interval, period 1.
	2330	Collect samples.
7	0100	Increase feed to 45.4 kg (100 lb)/h; adjust conditions given for phase 2, period 2, in Table 3.
	0500	Collect samples.
	0630	Begin second sample interval, period 2.
	1030	Collect samples.
	1100	Adjust operating conditions.
	1200	Begin phase 2, period 3.
	1600	Collect samples.
	1730	Begin second sample interval, period 3.
	2130	Collect samples.
	2200	Adjust operating conditions.
	2300	Begin phase 2, period 4.
8	0300	Collect samples.
	0530	Begin second sample interval, period 4.
	0930	Collect samples.
	1030	Begin cooldown.
9	1200	Complete shutdown.
10	0900	Open lower chamber, clean and collect additional ash samples.

TABLE 2. PLANNED OPERATING CONDITIONS

OFFGAS SYSTEM CONTROL SETTINGS	
Venturi ΔP	14.9 kPa (60 in. W.G.)
Venturi solution flow	30.3 L/min (8 gal/min)
Absorber solution flow	18.7 L/min (5 gal/min)
Quench liquid flow	60.6 L/min (15.7 gal/min)
Solution pH (absorber, quench, and venturi)	8 to 9
Condenser gas-phase temperature decrease	17°C (30°F) normal*
Superheater temperature increase	17°C (30°F)
HEPA filter ΔP	0.02 to 0.42 kPa (0.005 to 0.1 in. W.G.) normal*

* Normal values - not controlled.

INCINERATOR			
Lower chamber pressure	0.49 kPa below ambient (–2 in. W.G.)		
Steam to lower chamber	2.3 kg (5 lb)/h (for cooling steam nozzles)		
Phase 1:	Baseline with untreated wood		
Period 1:	Feed 45.4 kg (100 lb)/h		
	Upper chamber temp 1010°C (1850°F)	O_2	10%
	Lower chamber temp 927°C (1700°F)	O_2	5% min.
Period 2:	Feed 45.4 kg/h		
	Upper chamber temp 1093°C (2000°F)	O_2	10%
	Lower chamber temp 1010°C (1850°F)	O_2	5% min.
Phase 2:	Variation of temperature, treated wood, feed rate, excess air		
Period 1:	Feed 27.2 kg (60 lb)/h		
	Upper chamber temp 1204°C (2200°F)	O_2	10%
	Lower chamber temp 927°C (1700°F)	O_2	5% min.
Period 2:	Feed 45.4 kg (100 lb)/h		
	Upper chamber temp 1204°C (2200°F)	O_2	10%
	Lower chamber temp 1038°C (1900°F)	O_2	2% min.
Period 3:	Feed 45.4 kg (100 lb)/h		
	Upper chamber temp 1204°C (2200°F)	O_2	10%
	Lower chamber temp 927°C (1700°F)	O_2	0%
Period 4:	Feed 45.4 kg (100 lb)/h		
	Upper chamber temp 1093°C (2000°F)	O_2	10%
	Lower chamber temp 927°C (1700°F)	O_2	5% min.

TABLE 3. PCP RUN CHRONOLOGY

Test day	Time	Event
1	0815	Began startup.
	0915	Auxiliary generator - 400.8 h, fuel tank 11,355 L (1000 gal) (full).
	1000	Lost probe tip from interchamber probe into lower chamber (quartz liner and tip).
	1430	Replaced caps on sample probe ports.
	1635	Programmable set point malfunctioned. Pen was hanging up on drum.
	1755	Lost lower burner several times. Relit.
	2000	Programmable set point pen hanging up.
	2030	Lower burner flame out when secondary air blower started up. Combustion air to gas ratio controller setting changed to 475.
	2100	Both burners off on ID blower shutdown. Relit. Reset programmable set point.
2	0100	Lost lower burner several times. Reset ratio controller to 325.
	0200	Tempering steam on at 2.8 kg (6.2 lb)/h. Programmable set point not tracking. Went to manual control of heatup.
	0930	Inserted interchamber sampling probe. Turned on cooling water at 8 L (2 gal)/min after inserting probe. Quartz tube broke from thermal shock.
	1030	Interchamber probe broken. Undetermined amount of water flowed into lower chamber.
	1105	Cycled ash door to remove broken glass from probes. Noted water in GADOS. Water was drained and contained. No spread of contamination. Absorber liquid flow valve on manual.
	1445	Absorber sump high-level alarm. While draining absorber sump, venturi pressure drop cycled erratically. Lower chamber high-pressure alarm caused a controlled shutdown. Positive incinerator pressure necessitated shutdown of burners. Fast shutdown probably initiated by low HEPA filter pressure drop. Reduced liquid flow to absorber tower; changed liquid filters. Recovered system.
	1815	Relit lower burner. Back on manual heatup.
	2330	Absorber sump high-level alarm. Reduced absorber flow. Low-level alarm. Increased flow.
3	0250	Tested interchamber sampler. Water was drawn into impingers showing that glass liner in probe had broken. Began replacement of glass tube with SS tube.
4	0125	New probe liner installed.
	0215	Fed first box of untreated wood, period 1 of phase 1.
	0218	Started first sample period.
	0220	Second box fed as load timer was set to zero. Burners shut down due to low HEPA pressure drop. Recovered system.
	0230	Lowered feed rate to 27.2 kg (60 lb)/h. Dropped lower chamber temperature controller to 900°C (1652°F).
	0330	Raised lower chamber temperature controller to 927°C (1700°F).

TABLE 3. (cont)

Test day	Time	Event
4	0600	Guillotine door stuck in up position during charging cycle. Main ram cycled. Lowered door by manually operating relays. Cycled ram several times without problems.
	0618	Finished sample period.
	1055	Cycled ram four times. No problems.
	1300	Loaded box.
	1303	Started second sample period, phase 1, period 1.
	1700	Finished second sample period, phase 1, period 1.
	1900	Transferred ash. Large amount of water mixed with ash after transfer due to earlier probe failures.
	2040	Fast shutdown caused by high chamber pressure while trying to obtain 10% O_2 in upper chamber. Decided to run at lower O_2 level.
5	0040	Started first sample period, phase 1, period 2.
	0440	Ended first sample period, phase 1, period 2.
	0900	Changed quench filters.
	0945	Started second sample period, phase 1, period 2.
	1345	Finished second sample period, phase 1, period 2. Started cooldown.
	2030	Shut off lower burner.
	2130	Shut off upper burner.
6	0055	Shut off secondary air blower.
	0330	Changed one sample pump and gauges on the carbon bed sample system.
	1800	Opened lower chamber door. Little ash present. Took photographs. Vacuumed out all ash with wand.
	2000	Ash transferred, lower chamber buttoned up. Began startup.
7	1005	Fed first box, phase 2, period 4. Burner flame out due to burner being at high fire when charging. Raised underfire air to 75.
	1025	Feed rate increased to 36.3 kg (80 lb)/h.
	1125	Started first sample, phase 2, period 1.
	1300	Box stuck inside ram; ram hung up on a piece of wood from box. Box burnt in ram with guillotine door in up position. Subsequent feed cycles satisfactory.
	1545	Finished first gas sample, phase 2, period 1.
	1825	Started second gas sample, phase 2, period 1.
	2225	Finished second sample, phase 2, period 1.
	2245	Low fuel level light on at auxiliary generator.
	2250	Auxiliary generator down.
8	0100	Auxiliary generator fuel arrived.
	0200	Fuel oil transfer pump impeller damaged (apparently broken up). Replaced pump.
	0340	Found caustic pump leaking.
	0430	Replaced caustic pump.
	0510	Resumed feed at 27.2 kg (60 lb)/h.
	0540	Burner flame out on first two feed cycles. Lower burner placed on manual control.

TABLE 3. (cont)

Test day	Time	Event
8	0550	Started first sample, phase 2, period 2.
	0950	Finished first sample, phase 2, period 2.
	1100	Started second sample, phase 2, period 2.
	1500	Finished second sample, phase 2, period 2.
	1530	Transferred ash.
	1600	Started first sample, phase 2, period 3.
	1720	Alarm indicated high lower chamber temperature.
	1750	Reset lower chamber high-temperature switch.
	1830	Switched to east bank of quench filters. Changed out west bank.
	2000	Finished first sample, phase 2, period 3.
	2240	Started second sample, phase 2, period 3.
9	0040	Lower burner flame out. Relit.
	0130	Additional loss of flame on lower burner when minimum stop was reduced.
	0240	Finished second gas sample, phase 2, period 3.
	0550	Replaced sample line for interchamber probe.
	0600	Started first sample, phase 2, period 4.
	1000	Finished first sample, phase 2, period 4.
	1200	Started second sample, phase 2, period 4.
	1340	Fast shutdown caused by high lower chamber temperature indication. Chart did not show rise in temperature.
	1430	Fast shutdown, same problem. Secured thermocouple leads which solved problem.
	1600	Finished second sample, phase 2, period 4. Began shutdown.

SECTION 7

TEST RUN

The test run, as completed, differed from both the conceptual test schedule (Table 1) and from the experimental design. The deviations, reasons for the deviations, and impact of the deviations are discussed in the following subsections.

7.1 OFFGAS SYSTEM CONTROL SETTINGS

No major problems were experienced with the offgas system operation or control during the test run.

A minor problem with the absorber liquid flow indicator resulted in lower than planned liquid flow to the absorber. The flow was maintained at 15.9 L (4 gal)/min through phase 1 of the test and 17.4 L (4.5 gal)/min through phase 2.

A problem with the caustic addition pump following the phase 1 test resulted in a process liquid pH of approximately 6.5 during period 1 of phase 2 tests. The pump was replaced and pH returned to the 8-9 range for the duration of the run.

All other offgas system parameters remained normal throughout the run, and no impacts were expected from the minor deviations noted.

7.2 INCINERATOR

The incinerator operated at a nominal pressure of 0.49 kPa below ambient (-2 in. W.G.) throughout the run except for normal upsets during waste charging operations. Negative pressure is required to assure containment of hazardous materials within the process. Some deviation was also noted with operation at 1205°C (2200°F) in the upper chamber but was not less than 0.37 kPa below ambient (-1.5 in. W.G.) during normal operation. This deviation was caused by loading the offgas system to capacity, which was a test objective. No adverse impact on sample data was expected from this deviation.

Other incineration conditions are discussed in the subsections dealing with individual test periods.

7.3 SYSTEM STARTUP

During startup of the incineration system, problems were discovered with the EPA-Method 5 sample probe in the hot-zone (interchamber) sample port. The probe was water jacketed and quartz glass lined with a quartz glass tip. Leakage occurred from the water jacket around the liner at the tip end of the probe. This leak resulted in thermal shock which fractured the quartz

glass tip. Several attempts to correct the problem failed. The probe liner was replaced with a stainless steel tube and probe tip. No probiems with the probe were encountered following the change.

7.4 TEST PHASE 1, PERIOD 1

Test phase 1, period 1 was initiated at 0218 h on test day 4. Shortly after the start of the initial sampling interval, a fast feed cycle caused a shutdown of both burners. Both burners were relit without incident. The feed rate was lowered from 45.4 kg (100 lb)/h to 27.2 kg (60 lb)/h to keep the lower chamber at the desired temperature. Feed was interrupted at 0600 h due to a malfunction in the guillotine door on the incinerator feed ram. Because of this problem, the lower chamber temperature fell to 705°C (1300°F). The problem was resolved at 0605 h and feed was resumed. The first sample interval was concluded at 0618 h. Sample interval conditions were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**27.2 kg/h (60 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**704°C min., 993°C max, 916°C avg* (1300°F min., 1820°F max, 1680°F avg)**
Afterburner Temp	**1010°C (1850°F)**	**860°C min.,** 1054°C max, 1010°C avg (1580°F min., 1930°F max, 1850°F avg)**
Lower Chamber O_2	**5% min.**	**6% avg**
Afterburner O_2	**10%**	**11% avg**

***Does not include period following guillotine door failure.**
****Due to temp drop on initial burner failure.**

The second sample interval of phase 1, period 1, began at 1303 h and proceeded without incident concluding at 1700 h. Sample interval conditions were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**27.2 kg/h (60 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**849°C min., 971°C max, 927°C avg (1560°F min., 1780°F max, 1700°F avg)**
Afterburner Temp	**1010°C (1850°F)**	**1000°C min., 1038°C max, 1010°C avg (1832°F min., 1900°F max, 1850°F avg)**
Lower Chamber O_2	**5% min.**	**6% avg**
Afterburner O_2	**10%**	**9% avg**

7.5 TEST PHASE 1, PERIOD 2

Following completion of period 1, variables were adjusted in preparation for the second set of operating conditions planned for phase 1 of the test. Sampling in the first interval was initiated on day 5 at 0040 h. During the course of the interval, the lower burner went out several times, but it was relit each time without difficulty. The main effect of burner failures is the entrainment of particulates in the offgas during relights. Temperature loss is minimal because

sufficient heat is released by the burning wood. The sample interval concluded at 0440 h. Operating conditions were

	Planned	Actual
Feed Rate	45.4 kg/h (100 lb/h)	45.5 kg/h (100 lb/h)
Lower Chamber Temp	1010°C (1850°F)	954°C min., 1054°C max, 1004°C avg (1750°F min., 1930°F max, 1840°F avg)
Afterburner Temp	1093°C (2000°F)	1071°C min., 1127°C max, 1093°C avg (1960°F min., 2060°F max, 2000°F avg)
Lower Chamber O_2	5% min.	6% avg
Afterburner O_2	10%	8% avg

The lower than planned O_2 content of the afterburner effluent was due to loading of the offgas system and resulting limitation on secondary air input.

The second sample interval of phase 1, period 2, was started at 0945 h on day 5 and continued through 1345 h. Other than several flame failures on the lower burner, due to problems with the air/fuel-gas ratio control, no problems were encountered with incinerator operation in this interval. Conditions were as follows.

	Planned	Actual
Feed Rate	45.4 kg/h (100 lb/h)	38.6 kg/h (85 lb/h)
Lower Chamber Temp	1010°C (1850°F)	954°C min., 1043°C max, 1000°C avg (1750°F min., 1910°F max, 1832°F avg)
Afterburner Temp	1093°C (2000°F)	1077°C min., 1121°C max, 1093°C avg (1970°F min., 2050°F max, 2000°F avg)
Lower Chamber O_2	5% min.	6% avg
Afterburner O_2	10%	8% avg

7.6 INTERIM BETWEEN TEST PHASES

At the conclusion of phase 1, period 2, the incinerator was put into a controlled shutdown to cool the unit and to remove ash resulting from burning the untreated wood. During the downtime, adjustments and repairs were made to facilitate phase 2 operations. Shutdown and restart proceeded smoothly.

7.7 PHASE 2, PERIOD 1

This test was originally scheduled as phase 2, period 4. The schedule was changed to provide a more orderly progression in temperatures. This rescheduling also delayed period 3 until the end of the test. This shift was desirable because period 3 had the most potential for fouling equipment, sample trains, and lines.

Phase 2, period 1, was initiated on day 7 at 1125 h. Operations were normal until approximately 1300 h. At that time, a feed box jammed in the side ram and tore open as it entered the main feed ram housing. The main ram was blocked by a piece of loose wood, and the box ignited in the ram housing with the incinerator guillotine door open. This resulted in a

lower burner shutdown and a significant decrease in lower chamber temperature. Underfire air was also reduced automatically. Several manually initiated cycles of the feed system cleared the problem, and operation returned to normal at 1330 h, although the lower chamber temperature remained low until 1345 h. The impact of this problem, if any, would be reflected in the Method 5 probe hot-zone sample for this interval. The net result of the problem was low temperature and low excess air in the lower chamber for a prolonged period, causing low destruction efficiency for PCP and related compounds. The sample interval concluded at 1545 h.

The afterburner functioned normally throughout the disruption so that adverse effects were not expected in the hot offgas sample.

Nominal conditions, including the upset period, for this sample interval were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**36.3 kg/h (80 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**788°C min.,* 1000°C max, 918°C avg*** **(1450°F min., 1832°F max, 1648°F avg)**
Afterburner Temp	**1093°C (2000°F)**	**1077°C min., 1121°C max, 1099°C avg** **(1970°F min., 2050°F max, 2010°F avg)**
Lower Chamber O_2	**5% min.**	**4% avg***
Afterburner O_2	**10%**	**8% avg**

***Includes upset period.**

The second sample interval of phase 2, period 1, began at 1825 h on day 7 and was completed at 2225 h with no operational problems. An error in interpreting the run-plan change resulted in a high lower chamber temperature. Interval conditions were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**40.9 kg/h (90 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**927°C m[illegible] 1060°C max, 1002°C avg** **(1700°F [illegible], 1940°F max, 1836°F avg)**
Afterburner Temp	**1093°C (2000°F)**	**1027°C min., 1138°C max, 1104°C avg** **(1880°F min., 2080°F max, 2020°F avg)**
Lower Chamber O_2	**5% min.**	**5% avg**
Afterburner O_2	**10%**	**7.8% avg**

The afterburner oxygen content continued to be low as a result of limitations on secondary air input due to loading on the offgas system.

7.8 PHASE 2, PERIOD 2

Phase 2, period 2, was originally scheduled as phase 2, period 1. The first sample interval was started on day 8 at 0550 h. The interval was completed without problems at 0950 h. Operating conditions during the interval were

	Planned	Actual
Feed Rate	**27.2 kg/h (60 lb/h)**	**29.5 kg/h (65 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**871°C min., 993°C max, 927°C avg (1600°F min., 1820°F max, 1700°F avg)**
Afterburner Temp	**1204°C (2200°F)**	**1188°C min., 1227°C max, 1202°C avg (2170°F min., 2240°F max, 2196°F avg)**
Lower Chamber O_2	**5% min.**	**5% avg**
Afterburner O_2	**10%**	**7.5% avg**

The second sample interval ran with no problems from 1100 h to 1500 h on day 8. Interval operating conditions were

	Planned	Actual
Feed Rate	**27.2 kg/h (60 lb/h)**	**31.8 kg/h (70 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**871°C min., 1010°C max, 938°C avg (1600°F min., 1850°F max, 1720°F avg)**
Afterburner Temp	**1204°C (2200°F)**	**1199°C min., 1232°C max, 1210°C avg (2190°F min., 2250°F max, 2210°F avg)**
Lower Chamber O_2	**5% min.**	**6% avg**
Afterburner O_2	**10%**	**7.0% avg**

7.9 PHASE 2, PERIOD 3

Phase 2, period 3, was originally scheduled as phase 2, period 2. The first sample interval was started at 1600 h on day 8 and continued with no problems until 2000 h. Interval operating conditions were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**40.9 kg/h (90 lb/h)**
Lower Chamber Temp	**1038°C (1900°F)**	**927°C min., 1071°C max, 1027°C avg (1700°F min., 1960°F max, 1880°F avg)**
Afterburner Temp	**1204°C (2200°F)**	**1188°C min., 1238°C max, 1204°C avg (2170°F min., 2260°F max, 2200°F avg)**
Lower Chamber O_2	**5% min.**	**5% avg**
Afterburner O_2	**10%**	**6.5% avg**

The second sample interval ran from 2240 h on day 8 to 0240 h on day 9. The only abnormal occurrences were three flame failures on the lower burner. Since the wood feed rate was adequate to supply the required heat to the lower chamber, no impact on sample results was expected. Conditions during the sample interval were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**38.6 kg/h (85 lb/h)**
Lower Chamber Temp	**1038°C (1900°F)**	**927°C min., 1088°C max, 1032°C avg* (1700°F min., 1990°F max, 1890°F avg)**
Afterburner Temp	**1204°C (2200°F)**	**1193°C min., 1239°C max, 1204°C avg (2180°F min., 2262°F max, 2200°F avg)**
Lower Chamber O_2	**5% min.**	**5% avg**
Afterburner O_2	**10%**	**6% avg**

***Includes minor upset time.**

7.10 PHASE 2, PERIOD 4

Phase 2, period 4, was originally scheduled as phase 2, period 3. This set of planned conditions was likely to result in the poorest combustion conditions and a high potential for fouled equipment and plugged sample lines; therefore, it was delayed to the last. The first sample interval was started at 0600 h on day 9 and was completed at 1000 h. No operational problems were encountered. Conditions during the first sample interval were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**29.5 kg/h (65 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**904°C min., 1027°C max, 954°C avg (1660°F min., 1880°F max, 1750°F avg)**
Afterburner Temp	**1204°C (2200°F)**	**1199°C min., 1232°C max, 1210°C avg (2190°F min., 2250°F max, 2210°F avg)**
Lower Chamber O_2	**0%**	**1% avg**
Afterburner O_2	**10%**	**7.5% avg**

The second sample interval ran from 1200 h to 1600 h on day 9. At 1340 h, a false lower chamber temperature high alarm automatically initiated a fast shutdown. This problem recurred at 1430 h. The effect of these shutdowns was to increase the amount of uncombusted material carried out with the offgas and, therefore, into the samples. The sample results are conservative for nominal test conditions. Conditions, including the upsets, during the final sample interval were

	Planned	Actual
Feed Rate	**45.4 kg/h (100 lb/h)**	**27.2 kg/h (60 lb/h)**
Lower Chamber Temp	**927°C (1700°F)**	**827°C min.,* 1000°C max, 916°C avg* (1520°F min., 1832°F max, 1680°F avg)**
Afterburner Temp	**1204°C (2200°F)**	**1021°C min.,* 1227°C max, 1174°C avg* (1870°F min., 2240°F max, 2150°F avg)**
Lower Chamber O_2	**0%**	**0.5% avg**
Afterburner O_2	**10%**	**8.0% avg**

***Includes upset time.**

7.11 TEST RUN SUMMARY

A summary of the actual operating conditions for all sample intervals is given in Figure 10.

Calculations and data summaries include combustion and residence time calculations in Appendix B, destruction efficiency calculations in Appendix C, field data summaries in Appendix D, combustion efficiency calculations and offgas composition data in Appendix E, and sample and offgas flow calculations in Appendix F.

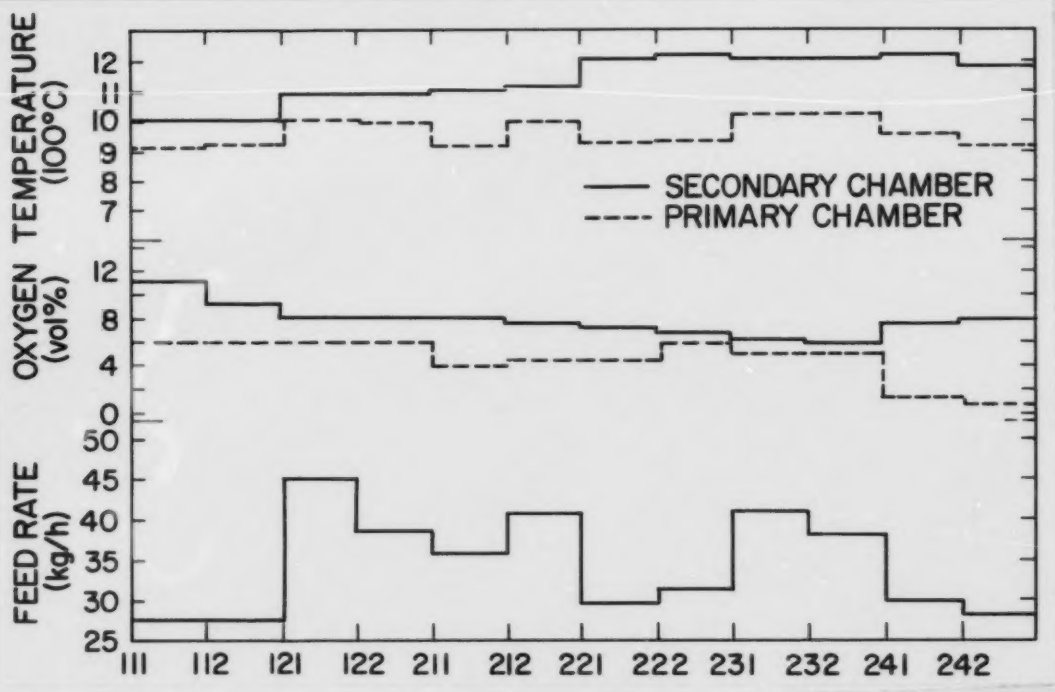


Figure 10. Average operating conditions during test interval abc (a = test phase, b = test period, c = sample interval).

SECTION 8

PROCEDURES AND ANALYTICAL RESULTS

This section details procedures for sample preparation, analytical procedures used by Los Alamos and SWRI, and results of analyses. The procedures were developed or modified for use in this program through consideration of special requirements and accepted practices.[1-4, 7-13]

8.1 SAMPLE PREPARATION

After completion of each sample interval during the PCP-treated wood test burn, the samples were prepared for analysis using procedures that depended on the sample type. All samples were refrigerated when not being analyzed.

8.1.1 Impinger Solvents

Impinger solvents were separated from moisture condensed in the sample trains. Water content was measured and recorded and the water discarded. The impinger catch and probe wash solvents were combined for a single sample from each train.

Analyses at Los Alamos were performed on "as received" and concentrated samples. Sample concentration was performed by vacuum drying a 25-mL sample at 40°C (105°F) and taking up the residue in 500 μL of 1:1 iso-octane:toluene containing 25 ppm 1-fluoro-2, 4 dinitrobenzene as an internal standard. Triplicate spiked samples (1 ppm PCP) were simultaneously concentrated by the above method with an average recovery of 70%.

Analyses at SWRI were done on sample concentrates prepared by reducing 30-mL samples to 0.5 mL by evaporation under a gentle stream of chromatographic-grade nitrogen and adding d_8-naphthalene, d_{10}-anthracene, and d_{12}-chrysene as internal standards. Final concentrations contained 51, 44, and 39 ng/μL of internal standards, respectively.

8.1.2 Sorbent Traps

Sorbent traps (XAD-2®) from the offgas sample trains were labeled and capped immediately after removal from the system. In preparation for analysis, the sorbent material was transferred to a Soxhlet extractor and extracted with 200 mL toluene for 20 h achieving a minimum of 80 cycles. The extract was then submitted for analysis.

8.1.3 Silica Gel

Silica gel from the moisture traps was weighed to determine moisture content and then stored. No additional analysis is planned.

8.1.4 Aqueous Scrub Solutions and Filters

Samples of the aqueous scrub solutions were extracted with toluene in three contacts using fresh solvent in each step. Extracts were combined prior to submitting the material to the laboratory. Scrub liquid filters did not have sufficient particles on them to provide a meaningful sample.

8.1.5 Ash

Ash samples were bagged in individual packages after phase 1 and after each period of phase 2 of the test. A measured quantity of ash was placed in a Soxhlet extractor and extracted with 200 mL toluene for 20 h achieving a minimum of 80 cycles. The solvent was then collected and submitted for anlaysis.

8.1.6 Wood

Samples of wood were taken at random from each packing case of shredded ammunition crates. The wood was reduced to splinters, blended, and divided into two samples. Each sample of the wood was placed in a Soxhlet extractor and extracted with toluene at 102°C for 24 h. These extracts were then analyzed to provide information on the PCP content (0.106 wt%) of the feed. These extracts were also analyzed for the target compounds listed in Appendix G, Table 1. None of these compounds, including TCDDs and TCDFs, were detected.

8.2. ANALYTICAL PROCEDURES

When submitted for analysis, all samples consisted of a solvent with unknown amounts of the target compounds in solution. The solvent was either toluene or a toluene: iso-octane mixture. The primary target compounds were pentachlorphenol, 2, 3, 7, 8-tetrachlorodibenzo-p-dioxin, and 2, 3, 7, 8-tetrachlorodibenzofuran.

Analyses were performed by the Los Alamos National Laboratory Industrial Hygiene Group H-5 Analytical Section with some sample splits analyzed by SWRI. The following subsections discuss procedures used by both laboratories in performing the analyses.

8.2.1. Los Alamos Procedures

Samples were analyzed in both the "as received" and concentrated conditions using several procedures. The "as received" samples were filtered to remove any particles entrained in the solvents. Concentrations were obtained by evaporating 25-mL samples at 40°C (104°F) under vacuum and taking up the residue in 500 μL of 1:1 toluene:iso-octane containing 25 ppm 1-fluoro-2, 4-dinitrobenzene as an internal standard.

Three techniques were used in analytical work at Los Alamos. They were gas chromatography/mass spectrometry (GC/MS), gas chromatography/electron capture detection (GC/ECD), and high-performance liquid chromatography (HPLC).

8.2.1.1 Qualitative Analysis by GC/MS—

Filtered samples were analyzed on a Hewlett-Packard 5992 GC/MS Data System adapted with a splitless capillary injection system and capillary restriction interface. This procedure is a combination of a high-resolution GC and a low-resolution MS. The column was a 30-m (98-ft) WCOT SE-54 fused silica capillary. Helium carrier gas at 165.5 kPa (24 psi) produced a 1.2-mL/min flow. The column oven was temperature programmed from 100°C (212°F) to 250°C (480°F) at 16°C (60°F)/min and held at 250°C (480°F) for a total of 60 min. Injector temperature was 270°C (520°F). The instrument was tuned daily by the Hewlett-Packard **AUTOTUNE®** program. Total ion signal over the range 20 to 600 amu was scanned at 380 amu/s. Simultaneously, the selected ion signal at 266 amu, corresponding to the most intense ion in the mass spectrum of PCP, was monitored.

Under these conditions and for a 2-μL injection volume and a 30-s injection time, the 95% confidence detection limit for PCP was 20 ppm.

8.2.1.2 Quantitative analysis by HPLC—

A Micrometritics 7000B HPLC was used with a 30-cm PARTISIL® PXCS 10/25 ODS-2 column. It was operated with constant carrier solvent composition at 1.5 mL/min with a solvent of 99% cyclohexane, 0.97% acetic acid, and 0.03% acetic anhydride. The UV detector was set at a wavelength of 300 nm to minimize absorption by the toluene solvent. The injection volume was 90 μL. The internal standard used was 1-fluoro-2, 4-dinitrobenzene.

For this system under these conditions, the 95% confidence detection limit was 0.5 ppm PCP, but only 10 ppm TCDD or TCDF. In the X50 concentrated samples, the 10-ppm detection limit corresponds to 0.2 ppm in the original samples.

The HPLC method was developed for back-up use due to early problems encountered with GC/ECD methods. It is discussed here only for information. The results were used for preliminary evaluation of the test, but HPLC was not the primary technique for analysis.

8.2.1.3 Quantitative Analysis by GC/ECD—

GC using an ECD was the method chosen for quantitative analysis of the test burn samples. However, a great deal of difficulty was encountered in developing suitable procedures.

Three different GCs were used in attempts to develop a satisfactory analytical procedure. The first was a Perkin-Elmer 900 GC with a Tracor ^{63}Ni ECD. The second was a Hewlett-Packard 5710 with a Valco Model 140B wide-range ECD. The third was a Varian 2800 GC with the same Valco ECD. The Perkin-Elmer and Varian chromatographs are set up to take only packed columns, whereas the Hewlett-Packard will take only capillary columns.

A large number of columns and conditions were tried in an attempt to obtain a satisfactory separation of the components of the samples. Among the columns tried were

3% OV-1 on Supelcoport;
3% OV-101 on Supelcoport;
3% OV-101 + 1 H_3PO_4 on Supelcoport;
3% OV-225 on Chromosorb G AW DMCS;
1% SP-1240 DA on Supelcoport;

1%	DEGS-PS on Supelcoport;
	Tenax GC;
	SE-54 WCOT capillary;
	Superpack-20M
	Chromosorb 102;
	Porapak P;
5%	Dexil 300 on Anakrom ABS;
3%	OV-17 on Chromosorb G AW DMCS;
3%	SE-30 on Saraport 30; and
0.3%	SP10000 + 0.3% H_3PO_4 on Carbopak A.

With most columns, the PCP peak was extremely broad, even at the maximum operating temperatures of the column. Such broad peaks led to long analysis time, poor resolutions, and poor detection limits.

Pentachlorophenol is a highly acidic, low-volatility compound which tends to decompose near its boiling point. This high acidity, combined with the high temperatures necessary to vaporize the compound and get it through a column, caused rapid degradation of the columns. In some cases, a column would appear to give acceptable results initially, but after the first few injections of standards, the results would deteriorate rapidly to become totally unsatisfactory. In other cases, the PCP would decompose or react to give more than one peak for standards which gave a single, sharp peak of the appropriate size when examined by HPLC.

To cut down on the acidity and increase the volatility of the analyte, attempts were made at derivatization of the PCP. The derivatizing agents trimethylchlorosilane (TMCS), N, O-Bis-(trimethylsilyl)-acrylamide (BSA), and N-trimethyl-silyl-imidazole (TSIM) were all tried.[14] Chromatographic results indicated that either the derivatization did not occur or the derivative was not stable under the chromatographic conditions.

The literature was not very helpful when it came to PCP analysis. A few recommended columns were found, but examples given were for 1000 to 2000 ppm PCP, and the methods would fail when tried at the 1 to 10 ppm levels.

The most successful analysis using GC/ECD during early efforts was with a Varian 2800 GC and a Valco Model 140B wide-range ECD. The 15-m × 0.3-cm (50-ft × 1/8-in.-o.d.) stainless steel column was packed with Tenax GC (60/80 mesh). Column temperature required was 280°C (540°F), at which point evidence of PCP decomposition was seen. Carrier gas flow was 38 mL/min 9:1 argon:methane. A 2-μL injection gave a detection limit of 0.5 ppm PCP. After a period of successful operation, this column also failed.

In preparing a new column, an attempt was made to improve performance by blocking adsorption sites via treatment with silanizing agents. The new column was a Supelco silane treated glass column, and the packing material was Supelco SP 1240 DA. The column was mounted in the Hewlett-Packard 5736 GC and thoroughly purged with carrier gas. It was then attached to the ^{63}Ni ECD. Instrument conditions for good determination of PCP with this system were subsequently found to be

Argon-Methane Carrier Flow Rate	**40 mL/min**
Injection Temperature	**250°C (480°F)**
Column Oven Temperature	**170°C (340°F)**
Detector Temperature	**300°C (570°F)**
Chromatograph Attenuation	**2**

PCP standards were prepared with a 10-ppm sample purchased from Regis Chemical Company* and by successive dilutions with toluene. Standard samples were prepared containing 1.0, 0.10, and 0.01 ppm PCP. The detection limit was 0.015 ppm for a 2-μL injection and the standard curve is reasonably linear to at least 10 ppm. Detection limits for TCDD and TCDF were determined to be 1 ppb and 5 ppb, respectively.

8.2.2. Southwest Research Institute Procedures

Samples were reduced in volume from 30 mL to 0.5 mL under a gentle stream of chromatographic-grade nitrogen. The internal standards d_8-chrysene were added to the concentrated extracts prior to injection; the final concentrations of the internal standards were 51, 44, and 39 ng/μL, respectively.

The instrument used for these analyses was a Finnigan 3623 quadrapole MS equipped with an INCOS® data system and a Tracor 560 GC. The fused silica capillary column is threaded through a heated conduit into the MS ion source.

Extracted ion current profiling was used to search for the following compounds in each total ion current (TIC) run:

chlorophenols—mono through pentachloro;
chlorobenzenes—di through hexachloro;
chlorodibenzodioxins—mono through octachloro; and
chlorodibenzofurans—mono through octachloro.

The following instrument operating conditions were used for the TIC runs.

GC	
Column	**15-m (49-ft) × 0.25-mm (0.01-in.) fused silica capillary**
Phase	**SE-54; 1.0 film**
Carrier gas	**He at 40 cm (16 in.)/s**
Temperature program	**64°C (147°F) for 1 min then +10°C (50°F)/min up to 300°C (572°F)**
Injector temperature	**280°C (540°F)**
Splitless injection	**---**

MS	
Electron energy	**70 eV**
Scan rate	**1 s/scan**
Mass resolution	**0.3 m/e**

*Regis Chemical Co., 8210 Austin Ave., Morton Grove, IL 60053.

Extracts were re-analyzed with the MS in the selected ion monitor (SIM) mode; target compounds were pentachlorophenol (m/e 266, 268) tetrachlorodibenzodioxin (m/e 320, 322), and tetrachlorodibenzofuran (m/e 304, 306). Other instrument operating conditions were identical to those listed for the TIC runs.

Detection limits for the target compounds are given in Table 1 of Appendix G (*note*: the reference to MID in Table 1 is an old nomenclature for SIM). The limits given are in micrograms per sample. The samples sent to SWRI were 30 mL each. Calculating the detection limits for the primary target compounds gives 0.17 ppm for PCP and 17 ppb for TCDD and TCDF.

8.3 ANALYTICAL RESULTS

Duplicate extracts from samples of the PCP-treated wood were analyzed at Los Alamos and found to contain 0.103% and 0.106% by weight of PCP to yield a chlorine content of 0.069 wt%, well below the 0.5 wt% level requiring chlorine scrubbing at 99% efficiency. The measured PCP content is the basis for the destruction efficiency calculation shown in Appendix C and the sample requirements calculations shown in Appendix B.

Results from analytical work on samples from the hot zone between the incinerator chambers and from the secondary chamber offgas are given in Table 4. These samples were the samples of primary interest in the test burn and were analyzed by both Los Alamos and SWRI. Variations in reported amounts of PCP in the samples by HPLC and GC/ECD are probably due to better component separation with the GC techniques.

No evidence of PCP, TCDD, or TCDF was found by Los Alamos in any scrub solution or ash extracts. The detection limits were 15 ppb for PCP, 1 ppb for TCDD, and 5 ppb for TCDF. Samples taken downstream of the offgas system both before and after the carbon bed showed no evidence of the target compounds. Criteria for detection included a 0.1-min retention time window with a response at 4 times noise level for PCP and a 0.05-min retention time window with 4 times noise level response for TCDD and TCDF.

Analysis by SWRI revealed no evidence of PCP, TCDD, or TCDF in the gas stream samples from the primary or secondary chambers or in extracts from the ash. Detection criteria concerning retention time window and signal-to-noise ratio for all target compounds were in accordance with EPA Method 625, Part 14, Qualitative and Quantitative Determinations.

TABLE 4. ANALYSIS OF PCP TEST SAMPLES

	Sample from			Results* (in sample)						
				by GC/ECD			by HPLC	by GC/MS		
Sample**	Phase	Period	Interval	PCP	TCDD	TCDF	PCP	PCP	TCDD	TCDF
HZ	1	1	1	ND†	ND	ND	ND	ND	ND	ND
HZ	1	1	2	ND	ND	ND	ND	ND	ND	ND
HZ	1	2	1	ND	ND	ND	ND	ND	ND	ND
HZ	1	2	2	ND	ND	ND	ND	ND	ND	ND
HZ	2	1	1	ND	ND	ND	1.64 ppm	ND	ND	ND
HZ	2	1	2	ND	ND	ND	ND	ND	ND	ND
HZ	2	2	1	ND	ND	ND	ND	ND	ND	ND
HZ	2	2	2	ND	ND	ND	ND	ND	ND	ND
HZ	2	3	1	ND	ND	ND	ND	ND	ND	ND
HZ	2	3	2	ND	ND	ND	ND	ND	ND	ND
HZ	2	4	1	ND	ND	ND	0.41 ppm	ND	ND	ND
HZ	2	4	2	ND	ND	ND	5.09 ppm	ND	ND	ND
HZ	individual samples from all test intervals			ND	ND	ND	ND	ND	ND	ND
Ash	individual samples from phase 1 and each phase 2 period			ND	ND	ND	ND	ND	ND	ND
Scrub liquids	composite of all phase 2 periods			ND	ND	ND	ND	---††	---	---
HZ XAD-2	2	2	1	ND	ND	ND	ND	---	---	---
OG XAD-2	2	2	1	ND	ND	ND	ND	---	---	---
HZ XAD-2	2	4	1	ND	ND	ND	ND	---	---	---
OG XAD-2	2	4	1	ND	ND	ND	ND	---	---	---
HZ XAD-2	2	4	2	ND	ND	ND	ND	---	---	---
OG XAD-2	2	4	2	ND	ND	ND	ND	---	---	---
HZ XAD-2	composite of other phase 2			ND	ND	ND	ND	---	---	---
OG XAD-2	composite of other phase 2			ND	ND	ND	ND	---	---	---

*GC/ECD and HPLC by Los Alamos.
GC/MS by Southwest Research Institute.

**Sample HZ = Hot zone between primary and secondary chambers;
OG = Offgas (secondary chamber effluent);
XAD-2 = Sorbent polymer extracts.

†ND = Below detection limits which are:
15 ppb for PCP by CG/ECD (Los Alamos),
1 ppb for TCDD by GC/ECD (Los Alamos),
5 ppb TCDF by GC/ECD (Los Alamos),
10 ppb for PCP by HPLC (Los Alamos),
170 ppb for PCP by GC/MS (Southwest Research Institute),
17 ppb for TCDD and TCDF by GC/MS (Southwest Research Institute).

††--- indicates not analyzed.

SECTION 9

DISCUSSION OF RESULTS

In the results of offgas sample analyses shown in Table 4, an apparent discrepancy exists between the results by HPLC and by GC/ECD techniques. This difference is most likely due to a non-PCP compound with a similar retention time in the HPLC system being reported as PCP. The HPLC work was done to provide preliminary data while problems with the GC/ECD procedure and equipment were resolved. No evidence of TCDF or TCDD was found in any sample by any of the analytical methods. The SWRI results, attached as Appendix G, show evidence in major peaks of some hydrocarbon components typical of wood burning. These peaks are not quantified.

The DE for PCP in the primary chamber was greater than 99.99% in all cases, based on the Los Alamos GC/ECD analytical data on concentrated samples. A sample DE calculation and results for all sample intervals are given in Appendix C. The actual DE for PCP during normal operation is expected to be much better than 99.99%, but the low concentration of PCP on the feed material and the difficult analysis for low levels of PCP in the offgas have precluded verification of a higher efficiency.

Results indicate that PCP-treated wood can be incinerated at controlled conditions so as to achieve >99.9% CE (>99.99% DE) without producing detectable levels of TCDD or TCDF. To achieve this, conditions in the primary chamber varied between approximately 910°C (1670°F) and 1025°C (1877°F) with oxygen concentrations in the chamber between 0.5% and 6.0%. Retention time for the gas phase in the primary chamber varied between 2.5 and 3.5 s based on effective use of 66.6% of the primary (lower) chamber volume. The remainder of the volume is considered ineffective because the burning waste pile extends about one-third of the hearth length.

It is the authors' judgement that proper operation of a single-chamber incinerator should also allow destruction of PCP-treated wood to the 99.99% DE level for PCP. Recommendations for such proper operation include:

- minimum chamber temperature >980°C (1800°F),
- gas retention time >2.5 s,
- avoidance of overfeeding that causes excessive smoking or low instantaneous excess oxygen levels,
- excess air >20% (oxygen >3% in offgas), and
- adequate time for ash burnout before adding fresh feed.

Any incinerator proposed for use should maintain these conditions to assure 99.99% DE of the PCP in the feed and to avoid formation of TCDD and TCDF.

REFERENCES

1. Lusten, Houwer, K. Olie, and O. Hutzinger, Chlorinated Dibenzo-P-Dioxins and Related Compounds in Incinerator Effluents. Chemosphere 9:501-522, 1980.

2. Ahling, B. and L. Johansson. Combustion Experiments Using Pentachlorophenol on a Pilot Scale and Full Scale. Chemosphere 7:425-436, 1977.

3. Rappe, C. and S. Marklund. Formation of Polychlorinated Dibenzo-P-Dioxins (PCDDs) and Dibenzofurans (PCDFs) By Burning or Heating Chlorophenates. Chemosphere 3:269-281, 1978.

4. The American Society of Mechanical Engineers Research Committee on Industrial and Municipal Wastes. Study on State-of-the-Art of Dioxin From Combustion Sources. New York, NY, 1981, 78 pp.

5. Warner, C. L., Ed. Final Safety Analysis Report for the Transuranic Contaminated Solid Waste Treatment Development Facility. Report LA-7971-MS, Los Alamos Scientific Laboratory, Los Alamos, NM, 1979.

6. Federal Register, January 23, 1981, pp. 7666-7690.

7. Hall, J. et al. Evaluation of PCP Destruction Efficiency in an Industrial Boiler. GCA Corporation, Technology Division, Bedford, MA, 1980, 19 pp.

8. Sano, T. L., D. R. Moore, and D. G. Ackerman. Emissions Testing During Incineration of PCBs at Rollins Environmental Services, Inc., Deer Park, Texas. TRW Incorporated, Environmental Engineering Division, Redondo Beach, CA, 1981, 130 pp.

9. Cavallaro, A. et al. Sampling Occurrence and Evaluation of PCDDs and PCDFs From Incinerated Solid Urban Waste. Chemosphere 9:611-621, 1980.

10. Ivanov, Z. and R. J. Magee, The Determinations of Trace Amounts of Chlorophenols by High-Performance Liquid Chromatography. Microchemical Journal 25:543-547, 1980.

11. diDomenico, L. et al. Analytical Techniques for 2, 3, 7, 8-Tetrachlorodibenzo-p-dioxin Detection in Environmental Samples after the Industrial Accident at Seveso. Analytical Chemistry 51(6): 735-740, 1979.

12. Nestrick, J., L. L. Lamparski, and R. H. Stehl. Synthesis and Identification of the 22 Tetrachlorodibenzo-p-dioxin Isomers by High Performance Liquid Chromatography and Gas Chromatography. Analytical Chemistry 51(13): 2273-2281, 1979.

13. McKinney, J. D., Ed. Environmental Health Chemistry, The Chemistry of Environmental Agents as Potential Human Hazards. Ann Arbor Science Publishers, Inc., Ann Arbor, MI, 1981, 656 pp.

14. The Regis Chemical Company. Regis Derivatization Guide. Morton Grove, IL, 1981.

APPENDIX A

QUALITY ASSURANCE

During this study efforts were made to validate all procedures and to provide assurance that the data obtained in the PCP test burn were reliable. Details of the QA considerations are given in the following sections.

A.1 METHOD 5 TRAINS

Calibration data were available for sample train pitot tubes and gas meters. Pitot and probe tips were carefully examined before each sample period to note any damage or other problems.

A.1.2 Glassware

All glassware was carefully cleaned by washing with soap and water followed by sequential rinses with de-ionized water, acetone, methanol, and methylene chloride before each use. Organic rinse materials were collected and checked for residues by the same methods used for normal samples.

Ground glass fittings were lightly greased only on outside edges to facilitate sealing and to avoid sample contamination.

A.1.3 Sample Bottles and Traps

All sample bottles were amber glass or foil-wrapped glass with TEFLON® seals in the lids. Sample traps (XAD-2® columns) were wrapped with aluminum foil to avoid UV exposure.

A.1.4 Reagents

All reagents used in sample collection and glassware rinsing after sample transfer were distilled-in-glass materials.

A.2 MONITORING EQUIPMENT

The incineration and offgas systems are equipped with extensive monitoring and control devices. Critical elements are checked regularly and calibrated to assure operability and reliability.

Prior to the run, on-line monitors, including analyzers for O_2, CO_2, and CO, were zeroed and calibrated using appropriate techniques. CO-CO_2 monitors were calibrated with a zero gas, full-span gas, and a mid-range gas and the calibration was checked daily during the burning of test materials.

A.3 SAMPLE LABELING AND CONTROL

All samples were identified by source, date, time, and collector. A unique sample identification number was assigned and a chain of custody was established for all samples when they were collected.

A.4 ANALYTICAL PROCEDURES

Assurance of the adequacy and reliability of analytical procedures is recognized as a major portion of the overall test program. A comprehensive program utilizing blanks and standards was developed to provide such assurance.

A.4.1 Blanks

Blanks were established for all types of samples and were included in sample sets subjected to analysis for the test run. Typical blanks for the PCP test run included

(a) ash from untreated wood burning,
(b) impinger solutions (unused solvents), and
(c) scrub solution makeup water.

Such blanks were given a sample identification number and were included with regular samples from the run.

A.4.2 Standards

Standards of chemicals of primary interest were obtained and used to spike samples for procedure verification. Standards were also used to calibrate analytical instruments, i.e., the GC/MS and GC/ECD systems. On the Los Alamos GC/ECD system, standards were run twice daily. Standard frequency at SWRI on the GC/MS was *at least* once and usually two or three times daily.

A.4.3 Duplicate Samples

The test plan included provisions for two sample periods at each set of operating conditions. While this did not provide a true duplicate sample, it did provide back-up samples in case some samples were lost or spoiled during analysis. Selected samples were split and, after the samples were shown to be free of radioactive contamination, they were sent to SWRI for analysis.

A.5 RECORDS

Sample records are maintained in a bound pre-numbered notebook. The record consists of the unique sample identification number, the sample type (impinger solution, ash, etc.), sample location, date, time, and initials of the operator who collected the sample. Analytical results were entered in the notebook when received.

Records accompanying each sample include only the sample identification number and type. Information such as sample location, operating conditions, time, etc., did not accompany the samples.

APPENDIX B

COMBUSTION, RESIDENCE TIME, AND SAMPLE REQUIREMENTS CALCULATIONS

The following preliminary calculations were performed prior to testing to predict offgas flows, residence times, and sampling time needed to verify a 99.99% destruction and removal efficiency for PCP and to detect TCDDs and TCDFs in the offgas stream. Actual residence time and flow calculations, based on operating data, are also included. Calculations are shown in the original units. Only the final results have been converted to metric units.

B.1 PRELIMINARY COMBUSTION CALCULATIONS

The PCP-treated wood is essentially pine with trace amounts of PCP added. For the purpose of the preliminary combustion calculations, the composition was assumed to be that of cellulose.

Heating value (pine, 12% moisture): 7960 Btu/lb .

Higher heating value (dry basis): $\frac{7960\ \text{Btu}}{\text{lb-wet}} \quad \frac{\text{lb-wet}}{(1\text{-}0.12)\ \text{lb-dry}} = 9066\ \text{Btu/lb}$.

Lower heating value (cellulose) at 60°F:

$$\frac{9066\ \text{Btu}}{\text{lb}} - \left(\frac{5\ \text{mole}\ H_2O}{\text{mole wood}} \times \frac{18\ \text{lb}\ H_2O}{\text{mole}\ H_2O} \times \frac{1060\ \text{Btu}}{\text{lb}\ H_2O} \times \frac{\text{mole wood}}{162\ \text{lb wood}} \right) = 8477\ \text{Btu/lb} .$$

Assumptions:
1. Lower chamber temperature is 2000°F.
2. Pine is equivalent to cellulose.
3. Feed rate is 100 lb/h.
4. Heat loss from the incinerator surface is 600 Btu/ft²·h.
5. Excess air is 50%.
6. Gas pilot contributes 10 000 Btu/h.

Reaction at stoichiometric air input:

$C_6H_{10}O_5 + 6O_2 + 22.8N_2 \rightarrow 6CO_2 + 5H_2O + 22.8N_2$.

Reaction at 50% excess air:

$C_6H_{10}O_5 + 9O_2 + 34.1N_2 \rightarrow 6CO_2 + 5H_2O + 14.37\ \text{air} + 22.7N_2$.

Mass and energy balance (per lb-mole wood feed):

Component	lb-moles Product	mol wt	$\Delta H_{2000\text{-}60}$ (Btu/lb)	Heat Req. (Btu)
CO_2	6	44.01	552.3	145 840
H_2O	5	18.02	1047.2	94 353
Air	14.37	28.84	530.2	219 731
N_2	22.73	28.00	540.0	343 678
Total	48.10			803 602

Heat required:

$$\frac{803\ 602\ \text{Btu/lb-mole}}{162\ \text{lb/lb-mole}} = 4960.5\ \text{Btu/lb wood burned}\ .$$

Heat release at 100 lb/h feed rate:

8477 Btu/lb × 100 lb/h =	847 700 Btu/h.
Heat duty to gases: 4960.5 Btu/lb wood × 100 lb wood/h =	−496 050
Surface loss: 600 Btu/ft²·h × 437 ft² surface =	−262 200
Input from natural gas pilot:	+10 000
Steam bleed (for nozzle cooling): 5 lb/h × 962.7 Btu/lb =	−4 813
Total:	94 637 Btu/h

Therefore, the 100 lb/h feed rate will load the incinerator (10% overload is within expected errors in assumptions).

B.2 PRELIMINARY RESIDENCE TIME CALCULATIONS

Offgas flow: (at 100 lb/h feed, 50% excess air, 2000°F lower chamber temp).

(a) from burner pilot (methane):

$$\frac{10\ 000\ \text{Btu/h}}{21\ 520\ \text{Btu/lb}} \times \frac{\text{lb-mole}}{16.04\ \text{lb}} = 0.03\ \text{lb-mole/h}\ ;$$

(b) from combustion products including excess air:

$$\frac{48.1\ \text{lb-mole prod.}}{\text{lb-mole wood}} \times \frac{\text{lb-mole wood}}{162\ \text{lb}} \times \frac{100\ \text{lb}}{\text{h}} = 29.66\ \text{lb-mole/h}\ ;$$

(c) from steam bleed:

$$\frac{5\ \text{lb steam}}{\text{h}} \times \frac{\text{lb-mole}}{18\ \text{lb}} = 0.28\ \text{lb-mole/h}\ .$$

Total: 29.97 lb-mole/h .

Temperature = 2200°F, chamber pressure = –2 in. W.G.

$$\frac{dV}{dt} = \left(\frac{RT}{p}\right)\left(\frac{dn}{dt}\right) = \frac{\left(\frac{10.73\ \text{psi}\cdot\text{ft}^3}{\text{lb-mole}\cdot{}^\circ\text{R}}\right)(2460{}^\circ\text{R})\left(\frac{29.97\ \text{lb-mole}}{\text{h}}\right)}{(11.3 - 2/27.07\ \text{psi})(60\ \text{min/h})} =$$

1174 ACFM (actual ft³ per minute) .

Volume of lower chamber: 108.1 ft³.

Effective volume of lower chamber (due to waste burning on first one-third of hearth):

108.1 ft³ × 0.66 = 72.0 ft³.

Predicted residence time:

$$\frac{(72.0\ \text{ft}^3)(60\ \text{s/min})}{1174\ \text{ACFM}} = 3.68\ \text{s}.$$

B.3 SAMPLE REQUIREMENT CALCULATIONS

B.3.1 PCP Detection

Assumptions:
1. 100 lb/h feed.
2. 250 SCFM offgas from lower chamber.
3. 0.5 SCFM sample rate.
4. 1-L sample from impinger catch.
5. 100% recovery of PCP in sample.
6. 0.5-ppm detection limit for sample.

Given: PCP concentration on wood = 0.106 wt% (based on an arithmetic average of two concentrations, one from each random wood sample).
Density of toluene solvent = 0.8 g/mL.

PCP feed rate: 100 lb/h × 0.00106 lb PCP/lb = 0.106 lb PCP/h.

PCP in offgas at 99.99% DRE: 0.106 lb PCP/h × 0.0001 = 1.06×10^{-5} lb/h.

Fraction total PCP in sample: $\frac{0.5\ \text{SCFM sample rate}}{250\ \text{SCFM offgas rate}} = 0.002.$

Rate into sample catch:

1.06×10^{-5} lb/h × 0.002 = 2.12×10^{-8} lb PCP/h (9.62×10^{-6} g/h) .

Mass of PCP required in sample with 50x concentration:

$$\frac{1000\ \text{mL}}{50} \times \frac{0.8\ \text{g}}{\text{mL}} \times \frac{0.5\ \mu\text{g}}{\text{g}} = 8\ \mu\text{g} .$$

Sampling time required: $\frac{8\ \mu g\ required}{9.62 \times 10^{-6}\ g/h} = 0.83\ h$.

B.3.2. TCDD/TCDF Detection

Assumptions as in Sec. B.3.1 except

1. 95% DRE for PCP.
2. 0.1% of remaining PCP is converted to TCDD or TCDF.
3. 100% of TCDD or TCDF goes to offgas.

Resulting TCDD or TCDF in offgas:

0.106 lb PCP/h × 5% remaining × 0.001 conversion = 5.35×10^{-6} lb/h .

Rate into sample catch:

5.3×10^{-9} lb/h × 0.002 sample fraction = 1.06×10^{-8} lb/h (4.81 μg/h).

Assuming the same detection limit, required sampling time is

$\frac{8\ \mu g}{4.81\ \mu g/h} = 1.66$ h.

Sample time selected was twice the required time rounded up to a whole hour:

1.66 h × 2 = 3.33 h.

Therefore, use 4-h sample period.

B.4. ACTUAL RESIDENCE TIME CALCULATIONS

Basis: Operating conditions from test phase 2, period 3, interval 1, during which the highest feed rate and lowest expected residence time were used for the actual residence time calculations. Oxygen content of the offgas was assumed correct as indicated by the on-stream continuous analyzers.

Data:		
	Chamber pressure	11.23 psia
	Lower chamber temperature	1880°F avg
	Lower chamber oxygen	5.0% avg
	Lower burner fuel-gas flow	5 SCFM
	Lower burner combustion air flow	45 SCFM
	Underfire air flow	92 SCFM
	Upper chamber temperature	2200°F avg
	Upper chamber oxygen	6.5% avg
	Upper burner fuel-gas flow	20.6 SCFM
	Upper burner combustion air flow	185 SCFM
	Secondary air flow	380 SCFM
	Wood feed rate	90 lb/h

B.4.1 Lower (Primary) Chamber Flow Calculations

Measured total air = 45 SCFM burner + 92 SCFM underfire = 137 SCFM.

Let P = % excess air/100 and assume wood feed has composition of cellulose; then

$$C_6H_{10}O_5 + (1 + P)6O_2 + (1 + P)22.6N_2 \rightarrow 6CO_2 + 5H_2O + P6O_2 + (1 + P)22.6N_2 \ .$$

Fuel gas flow to lower chamber = 5 SCFM (0.837 lb-mole/h).

Wood feed = 90 lb/h ÷ 162 lb/lb-mole = 0.555 lb-mole/h.

Mole fraction of fuels:

$f_{wood} = 0.555/(0.555 + 0.837) = 0.399$.

$f_{gas} = 1 - 0.399 = 0.601$.

Average fuel formula (wood + gas): $C_{2.995}H_{6.394}O_{1.995}$.

Mol wt: 74.254 .

General reaction per mole of feed:

$$C_{2.995}H_{6.394}O_{1.995} + 3.596\ (1 + P)O_2 + 13.528\ (1 + P)N_2 \rightarrow$$
$$2.995CO_2 + 3.197H_2O + (3.596P)O_2 + 13.528\ (1 + P)N_2 \ .$$

Solving for P, which corresponds to 5% oxygen in the offgas from the lower chamber, gives

P = 36.7% excess air in lower chamber.

Oxygen input to the lower chamber to meet 36.7% excess air was 6.84 lb-mole/h but measured flow was only 4.806 lb-mole/h; therefore in-leakage accounted for 2.042 lb-mole/h of the oxygen input.

The final offgas composition (leaving the lower chamber) was

Component	lb-moles/h	Mole Fraction
CO_2	4.172	0.113
H_2O from combustion	4.453	
H_2O from steam	0.555	0.136
O_2	1.839	0.050
N_2	25.736	0.700
Total	36.782	

The resulting flowrate from the lower chamber is

$$\frac{dV}{dt} = \frac{RT}{P}\frac{dn}{dt} = \frac{(10.73 \text{ psi·ft}^3/^\circ\text{R·lb-mole})(2340^\circ\text{R})(36.8 \text{ lb-mole/h})}{(11.23 \text{ psia})(60 \text{ min/h})} = 1370 \text{ ACFM} .$$

Residence time:

Assumes effective volume = 2/3 of lower chamber volume = 72 ft³.

$$t_{res} = \frac{V}{Q} = \frac{(72.0 \text{ ft}^3)(60 \text{ s/min})}{1370 \text{ ft}^3/\text{min}} = 3.15 \text{ s} .$$

B.4.2 Upper Chamber Flow Calculations

Assumptions:
1. negligible combustibles in offgas from lower chamber.
2. air input = measured flow minus 25 SCFM for the ram door, sightglass purges, etc.
3. no air in-leakage to upper chamber.
4. fuel gas is methane.

Combustible feed to upper chamber is then fuel gas only at the rate of 20.6 SCFM (3.441 lb-mole/h).

The combustion reaction formula for fuel gas with P% excess air is

$$CH_4 + (1 + P)2O_2 + (1 + P)(3.29)2N_2 \rightarrow CO_2 + 2H_2O + 2(P)O_2 + 6.58(1 + P)N_2 .$$

Solving for P, which corresponds to 6.5% O_2 in the offgas, gives

P = 62.1% excess air.

O_2 input from all sources to result in the 62.1% excess was 11.156 lb-mole/h, which carried with it 41.97 lb-mole/h N_2.

The resulting offgas flow from the upper chamber was then

Component	Lower Chamber	Upper Chamber	Total	Mole Fraction
CO_2	4.172	3.441	7.613	0.082
H_2O	5.008	6.882	11.890	0.127
O_2	1.839	4.270	6.110	0.066
N_2	25.763	41.970	67.730	0.725
Total			93.343	

Flowrate is then

$$\frac{dV}{dt} = \frac{RT}{P}\frac{dn}{dt} = \frac{(10.73 \text{ psi·ft}^3/^\circ\text{R·lb-mole})(2660^\circ\text{R})(93.34 \text{ lb-mole/h})}{(11.23 \text{ psia})(60 \text{ min/h})} = 3954 \text{ ACFM} .$$

Residence time, based on effective total volume (98.8 ft^3) of upper chamber (69.13 ft^3) plus hot duct (29.7 ft^3), is

$$t_{res} = \frac{V}{Q} = \frac{(98.8\ ft^3)(60\ s/min)}{3954\ ft^3/min} = 1.5\ s\ .$$

APPENDIX C

DESTRUCTION EFFICIENCY CALCULATIONS

The following calculations were used to evaluate the DE for PCP during the test burn:

$$DE_{PCP} = \frac{\text{PCP input} - \text{PCP emitted}}{\text{PCP input}} \times 100 \ .$$

PCP input = 0.106% (PCP on wood) × feed rate × sample duration.

PCP emitted = detection limit (since none was detected) back calculated to content in the offgas as follows:

solvent weight (g) × detection limit (g/g solvent) = potential content in sample (g).

From Appendix F:

$$\text{fraction of offgas sampled} = \frac{\text{sample gas volume (ft}^3\text{)}}{\text{offgas flow (ft}^3\text{/min)} \times \text{sample duration (min)}} \ .$$

$$\text{potential content of offgas (g)} = \frac{\text{potential content from sample (g)}}{\text{fraction sampled}} \ .$$

The potential content of the offgas is then assumed to be the PCP emitted.

Sample calculation: Test phase 2, period 4, interval 2. Hot-zone sample (lower chamber offgas)

solvent weight	704 g
detection limit	15×10^{-6} g/g solvent
sample gas volume	68.6 SCF
offgas flow	317.0 SCFM
sample duration	240 min
wood feed rate	27.2 kg/h

PCP input = 27.2×10^3 g/h × 0.106% PCP × 4 h = 115.328 g .

$$\text{PCP emitted} = \frac{15 \times 10^{-6}\text{ g/(g solvent)} \times 704\text{ (g solvent)}}{68.6\text{ SCF}/(317.0\text{ SCFM} \times 240\text{ min})} = 0.0117\text{ g} \ .$$

$$DE_{PCP} = \frac{115.328 - 0.0117}{115.328} \times 100 = 99.990\% \ .$$

The DE calculation was performed for all hot-zone samples as a measure of the effectiveness of a single-chamber incineration step for the disposal of PCP-treated wood (to model the proposed use of a single-chamber Korean incinerator for the disposal of such wood). The resulting DE values are included in the tabulation below. The detection limit used in these calculations was 15 ppb PCP in the original sample solvent by GC/ECD at Los Alamos.

TABLE C-1. DESTRUCTION EFFICIENCY SUMMARY

Test P-P-I*	Solvent (g)	Sample Volume (SCF)	Offgas Flow (SCFM)	Sample Duration (min)	Wood Feed Rate (kg/h)	DE (%)
2-1-1	640	65	296	240	36.3	99.993
2-1-2	688	79	290	240	40.9	99.995
2-2-1	600	91	286	240	29.5	99.995
2-2-2	648	97	298	240	31.8	99.995
2-3-1	688	89	290	240	40.9	99.995
2-3-2	704	61	283	240	38.6	99.993
2-4-1	688	86	281	240	29.5	99.994
2-4-2	704	69	317	240	27.2	99.990

*P-P-I = test Phase, Period, Interval.

APPENDIX D

FIELD DATA SUMMARY

The following data are a summary of the field data from the sample train control boxes and impingers used to calculate sample volumes and offgas flow rates. The data are given only in the original units.

Test Phase 1, Period 1, Interval 1
Barometric Pressure 22.03 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 112°F

Sample Location: HZ
Water Catch

Silica Gel	gross	252.8 g	
	tare	200.0 g	
	net		52.8 mL
Impinger	#1		15.8 mL
	#2		7.0 mL
	#3		1.0 mL
Cyclone			2.0 mL
Total Water			78.6 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	324.2	0.004	1640	75	70
2		0.004	1590	70	74
3		0.003	1630	62	72
4		0.003	1615	61	80
5		0.004	1615	65	82
6		0.003	1690	62	80
7		0.004	1710	60	89
8		0.003	1700	62	87
9		0.004	1720	60	89
10		0.004	1630	61	91
11		0.003	1650	62	91
12		0.002	1660	62	91
End	428.5				

Test Phase 1, Period 1, Interval 2
Barometric Pressure 23.09 in. Hg
Sample Zone Pressure 22.95 in. Hg
Ambient Temperature 115°F

Sample Location: HZ
Water Catch

Silica Gel	gross	250.3 g	
	tare	200.0 g	
	net		50.3 mL
Impinger	#1		25.0 mL
	#2		4.0 mL
	#3		- mL
Cyclone			- mL
Total Water			79.3 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	429.7	0.005	1675	70	70
2		0.005	1770	75	70
3		0.005	1790	85	80
4		0.005	1810	90	85
5		0.004	1800	100	90
6		0.004	1830	95	90
7		0.004	1870	100	95
8		0.004	1830	90	90
9		0.004	1820	90	90
10		0.004	1820	95	95
11		0.004	1800	100	100
12		0.004	1800	105	105
End	530.0				

Test Phase 1, Period 2, Interval 1
Barometric Pressure 23.09 in. Hg
Sample Zone Pressure 22.95 in. Hg
Ambient Temperature 115°F

Sample Location: HZ
Water Catch

Silica Gel	gross	252.5 g	
	tare	200.0 g	
	net		52.5 mL
Impinger	#1		5.2 mL
	#2		5.0 mL
	#3		- mL
Cyclone			15.0 mL
Total Water			77.7 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	530.6	0.005	1780	82	77
2		0.005	1840	86	83
3		0.005	1860	89	84
4		0.005	1847	91	88
5		0.006	1855	93	89
6		0.004	1839	94	91
7		0.006	1872	94	91
8		0.006	1826	96	90
9		0.005	1846	95	92
10		0.005	1852	96	92
11		0.005	1861	95	90
12		0.005	1823	95	89
End	537.9				

Test Phase 1, Period 2, Interval 2
Barometric Pressure 23.07 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 111°F

Sample Location: HZ
Water Catch

Silica Gel	gross	248.3 g	
	tare	200.0 g	
	net		46.3 mL
Impinger	#1		20.1 mL
	#2		- mL
	#3		- mL
Cyclone			15.2 mL
Total Water			83.6 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	639.2	0.005	1760	80	75
2		0.004	1775	82	76
3		0.004	1900	83	78
4		0.004	1820	85	80
5		0.005	1915	86	81
6		0.004	1810	86	81
7		0.005	1800	88	83
8		0.004	1850	89	83
9		0.005	1875	91	84
10		0.004	1875	90	85
11		0.005	1850	89	85
12		0.004	1835	89	85
End	750.6				

Test Phase 2, Period 1, Interval 1
Barometric Pressure 23.05 in. Hg
Sample Zone Pressure 22.91 in. Hg
Ambient Temperature 116°F

Sample Location: HZ
Water Catch

Silica Gel	gross	242.5 g	
	tare	200.0 g	
	net		42.5 mL
Impinger	#1		13.3 mL
	#2		- mL
	#3		- mL
Cyclone			12.7 mL
Total Water			68.5 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	759.5	0.005	1660	75	71
2		0.005	1675	82	79
3		0.004	1700	88	82
4		0.004	1780	91	83
5		0.005	1750	93	85
6		0.005	1780	94	85
7		0.006	1700	94	86
8		0.004	1750	96	87
9		0.005	1675	95	87
10		0.005	1725	95	87
11		0.005	1700	95	87
12		0.005	1655	95	87
End	847.2				

Test Phase 2, Period 1, Interval 2
Barometric Pressure 23.05 in. Hg
Sample Zone Pressure 22.91 in. Hg
Ambient Temperature 125°F

Sample Location: HZ
Water Catch

Silica Gel	gross	242.5 g	
	tare	200.0 g	
	net		42.5 mL
Impinger	#1		10.0 mL
	#2		7.6 mL
	#3		- mL
Cyclone			3.0 mL
Total Water			63.1 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	848.3	0.004	1620	81	76
2		0.005	1705	84	79
3		0.005	1815	85	81
4		0.006	1750	91	81
5		0.005	1710	93	83
6		0.005	1795	93	83
7		0.004	1820	94	84
8		0.005	1790	95	85
9		0.005	1805	93	86
10		0.004	1780	92	87
11		0.004	1690	91	86
12		0.004	1670	91	87
End	953.7				

Test Phase 2, Period 2, Interval 1
Barometric Pressure 23.09 in. Hg
Sample Zone Pressure 22.95 in. Hg
Ambient Temperature 118°F

Sample Location: HZ
Water Catch

Silica Gel	gross	209.3 g	
	tare	200.0 g	
	net		9.3 mL
Impinger	#1		22.6 mL
	#2		5.3 mL
	#3		- mL
Cyclone			14.7 mL
Total Water			51.9 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	851.7	0.004	1680	81	77
2		0.004	1809	84	77
3		0.003	1810	86	79
4		0.004	1710	88	82
5		0.005	1760	91	83
6		0.006	1780	93	85
7		0.005	1695	92	87
8		0.005	1725	92	87
9		0.005	1750	93	87
10		0.004	1690	93	86
11		0.004	1735	95	86
12		0.004	1680	95	86
End	972.2				

Test Phase 2, Period 2, Interval 2
Barometric Pressure 23.07 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 127°F

Sample Location: HZ
Water Catch

Silica Gel	gross	253.8 g	
	tare	200.0 g	
	net		53.8 mL
Impinger	#1		20.2 mL
	#2		1.8 mL
	#3		- mL
Cyclone			30.0 mL
Total Water			105.8 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	972.7	0.005	1625	82	78
2		0.004	1700	83	81
3		0.005	1850	88	84
4		0.005	1800	91	86
5		0.006	1755	93	87
6		0.006	1810	93	88
7		0.005	1850	95	88
8		0.005	1800	94	89
9		0.004	1775	94	91
10		0.004	1750	94	92
11		0.005	1750	95	92
12		0.006	1695	95	92
End	1103.5				

Test Phase 2, Period 3, Interval 1
Barometric Pressure 22.03 in. Hg
Sample Zone Pressure 22.89 in. Hg
Ambient Temperature 130°F

Sample Location: HZ
Water Catch

Silica Gel	gross	242.5 g	
	tare	200.0 g	
	net		42.5 mL
Impinger	#1		21.7 mL
	#2		6.2 mL
	#3		- mL
Cyclone			12.5 mL
Total Water			82.9 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	103.7	0.005	1775	75	77
2		0.005	1840	84	83
3		0.005	1900	89	86
4		0.005	1880	93	91
5		0.004	1920	96	92
6		0.004	1885	97	96
7		0.005	1910	97	95
8		0.005	1875	98	98
9		0.006	1885	99	96
10		0.006	1895	100	96
11		0.005	1850	100	95
12		0.004	1815	99	95
End	224.1				

Test Phase 2, Period 3, Interval 2
Barometric Pressure 23.04 in. Hg
Sample Zone Pressure 22.91 in. Hg
Ambient Temperature 125°F

Sample Location: HZ
Water Catch

Silica Gel	gross	227.9 g	
	tare	200.0 g	
	net		27.9 mL
Impinger	#1		12.2 mL
	#2		5.0 mL
	#3		- mL
Cyclone			11.6 mL
Total Water			56.7 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	224.7	0.005	1840	71	73
2		0.005	1880	75	74
3		0.004	1910	79	78
4		0.004	1885	82	81
5		0.004	1845	85	82
6		0.005	1915	88	83
7		0.006	1855	89	83
8		0.007	1835	88	83
9		0.005	1870	89	84
10		0.004	1865	89	84
11		0.004	1890	90	85
12		0.003	1805	90	86
End	305.6				

Test Phase 2, Period 4, Interval 1
Barometric Pressure 22.13 in. Hg
Sample Zone Pressure 22.99 in. Hg
Ambient Temperature 123°F

Sample Location: HZ
Water Catch

Silica Gel	gross	243.3 g	
	tare	200.0 g	
	net		43.3 mL
Impinger	#1		47.8 mL
	#2		12.3 mL
	#3		1.5 mL
Cyclone			27.1 mL
Total Water			132.0 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	305.7	0.006	1680	81	76
2		0.005	1740	82	81
3		0.004	1720	85	82
4		0.004	1790	91	88
5		0.004	1785	94	91
6		0.004	1825	97	95
7		0.004	1705	101	99
8		0.005	1785	103	100
9		0.005	1800	103	100
10		0.005	1785	104	100
11		0.005	1775	104	101
12		0.004	1695	104	101
End	421.9				

Test Phase 2, Period 4, Interval 2
Barometric Pressure 23.07 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 123°F

Sample Location: HZ
Water Catch

Silica Gel	gross	226.9 g	
	tare	200.0 g	
	net		26.9 mL
Impinger	#1		24.3 mL
	#2		12.1 mL
	#3		1.0 mL
Cyclone			14.6 mL
Total Water			78.9 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	422.3	0.005	1695	81	75
2		0.004	1748	84	77
3		0.008	1810	85	79
4		0.006	1795	88	80
5		0.005	1641	91	83
6		0.006	1695	89	83
7		0.007	1705	91	85
8		0.007	1638	92	85
9		0.006	1695	88	82
10		0.005	1710	89	82
11		0.005	1655	91	83
12		0.005	1630	91	82
End	513.7				

Test Phase 1, Period 1, Interval 1
Barometric Pressure 23.09 in. Hg
Sample Zone Pressure 22.95 in. Hg
Ambient Temperature 112°F

Sample Location: OG
Water Catch

Silica Gel	gross	252.2 g	
	tare	200.0 g	
	net		52.2 mL
Impinger	#1		25.5 mL
	#2		7.2 mL
	#3		- mL
Cyclone			20.3 mL
Total Water			105.2 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	490.1	0.07	1605	88	85
2		0.07	1686	91	84
3		0.08	1710	97	84
4		0.09	1711	98	87
5		0.08	1727	100	89
6		0.07	1718	101	90
7		0.07	1703	101	89
8		0.07	1696	100	89
9		0.07	1699	100	89
10		0.07	1701	100	89
End	613.6				

Test Phase 1, Period 1, Interval 2
Barometric Pressure 23.07 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 115°F

Sample Location: OG
Water Catch

Silica Gel	gross	245.3 g	
	tare	200.0 g	
	net		45.3 mL
Impinger	#1		28.3 mL
	#2		2.5 mL
	#3		- mL
Cyclone			15.2 mL
Total Water			91.3 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	613.9	0.06	1631	73	71
2		0.06	1633	79	76
3		0.06	1664	88	84
4		0.05	1681	94	91
5		0.06	1670	97	95
6		0.06	1669	99	97
7		0.05	1677	100	98
8		0.06	1700	101	99
9		0.06	1678	102	101
10		0.06	1673	101	100
End	713.2				

Test Phase 1, Period 2, Interval 1
Barometric Pressure 23.09 in. Hg
Sample Zone Pressure 22.95 in. Hg
Ambient Temperature 115°F

Sample Location: OG
Water Catch

Silica Gel	gross	223.3 g	
	tare	200.0 g	
	net		23.3 mL
Impinger	#1		7.7 mL
	#2		1.1 mL
	#3		- mL
Cyclone			- mL
Total Water			32.1 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	774.1	0.07	1718	71	70
2		0.08	1816	75	75
3		0.07	1804	80	80
4		0.07	1831	86	84
5		0.07	1810	88	85
6		0.08	1818	89	85
7		0.08	1810	89	85
8		0.07	1850	89	85
9		0.07	1865	88	84
10		0.07	1843	88	83
End	795.4				

Test Phase 1, Period 2, Interval 2
Barometric Pressure 23.07 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 111°F

Sample Location: OG
Water Catch

Silica Gel	gross	254.5 g	
	tare	200.0 g	
	net		54.5 mL
Impinger	#1		20.3 mL
	#2		6.5 mL
	#3		- mL
Cyclone			8.2 mL
Total Water			89.5 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	796.9	0.08	1755	74	65
2		0.07	1790	91	76
3		0.08	1857	102	86
4		0.08	1805	108	93
5		0.09	1846	112	98
6		0.09	1846	116	101
7		0.08	1821	116	103
8		0.07	1797	116	104
9		0.08	1802	119	106
10		0.07	1812	122	108
End	923.1				

Test Phase 2, Period 1, Interval 1
Barometric Pressure 23.05 in. Hg
Sample Zone Pressure 22.91 in. Hg
Ambient Temperature 116°F

Sample Location: OG
Water Catch

Silica Gel	gross	254.9 g	
	tare	200.0 g	
	net		54.9 mL
Impinger	#1		33.9 mL
	#2		1.3 mL
	#3		- mL
Cyclone			37.4 mL
Total Water			127.5 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	923.3	0.08	1747	65	62
2		0.09	1800	90	75
3		0.09	1855	103	88
4		0.10	1910	110	95
5		0.08	1864	114	101
6		0.08	1905	117	104
7		0.09	1850	119	105
8		0.08	1891	121	107
9		0.08	1852	121	107
10		0.07	1863	122	109
End	1006.1				

Test Phase 2, Period 1, Interval 2
Barometric Pressure 23.05 in. Hg
Sample Zone Pressure 22.91 in. Hg
Ambient Temperature 125°F

Sample Location: OG
Water Catch

Silica Gel	gross	256.0 g	
	tare	200.0 g	
	net		56.0 mL
Impinger	#1		21.4 mL
	#2		0.9 mL
	#3		- mL
Cyclone			28.7 mL
Total Water			107.0 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	7.7	0.08	1780	82	75
2		0.08	1805	103	88
3		0.09	1890	113	98
4		0.09	1889	118	104
5		0.09	1852	119	105
6		0.08	1837	119	107
7		0.08	1831	120	107
8		0.07	1864	120	107
9		0.07	1886	120	106
10		0.08	1855	119	106
End	121.1				

Test Phase 2, Period 2, Interval 1
Barometric Pressure 23.09 in. Hg
Sample Zone Pressure 22.95 in. Hg
Ambient Temperature 118°F

Sample Location: OG
Water Catch

Silica Gel	gross	253.3 g	
	tare	200.0 g	
	net		53.3 mL
Impinger	#1		29.7 mL
	#2		17.9 mL
	#3		1.0 mL
Cyclone			42.4 mL
Total Water			144.3 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	121.9	0.09	1829	80	70
2		0.09	1847	96	80
3		0.09	1908	110	94
4		0.09	1982	113	98
5		0.085	1991	118	103
6		0.095	1987	117	104
7		0.085	1982	119	105
8		0.095	1984	120	107
9		0.09	1966	119	106
10		0.09	1973	120	107
End	252.0				

Test Phase 2, Period 2, Interval 2
Barometric Pressure 23.07 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 127°F

Sample Location: OG
Water Catch

Silica Gel	gross	252.8 g	
	tare	200.0 g	
	net		52.8 mL
Impinger	#1		16.7 mL
	#2		- mL
	#3		- mL
Cyclone			48.3 mL
Total Water			117.8 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	252.4	0.09	1886	89	83
2		0.09	1914	93	87
3		0.10	1998	97	103
4		0.09	1947	99	107
5		0.10	1968	98	106
6		0.09	1995	99	106
7		0.09	1985	101	109
8		0.08	1992	102	112
9		0.09	1989	101	111
10		0.08	1992	103	105
End	355.9				

Test Phase 2, Period 3, Interval 1
Barometric Pressure 23.03 in. Hg
Sample Zone Pressure 22.89 in. Hg
Ambient Temperature 130°F

Sample Location: OG
Water Catch

Silica Gel	gross	254.0 g	
	tare	200.0 g	
	net		54.0 mL
Impinger	#1		37.4 mL
	#2		11.6 mL
	#3		3.8 mL
Cyclone			91.2 mL
Total Water			198.0 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	356.7	0.09	1874	100	106
2		0.09	2000	103	111
3		0.09	1999	104	106
4		0.09	2000	105	104
5		0.09	2010	104	103
6		0.08	2000	104	105
7		0.08	2010	105	106
8		0.08	2010	106	103
9		0.08	1965	106	103
10		0.07	1953	107	105
End	473.6				

Test Phase 2, Period 3, Interval 2
Barometric Pressure 23.05 in. Hg
Sample Zone Pressure 22.91 in. Hg
Ambient Temperature 125°F

Sample Location: OG
Water Catch

Silica Gel	gross	254.2 g	
	tare	200.0 g	
	net		54.2 mL
Impinger	#1		36.7 mL
	#2		31.3 mL
	#3		6.6 mL
Cyclone			93.4 mL
Total Water			222.2 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	479.0	0.08	1899	96	83
2		0.09	1965	109	94
3		0.09	2000	113	100
4		0.09	2000	115	102
5		0.08	1998	114	102
6		0.09	1996	114	101
7		0.08	2000	114	102
8		0.08	2000	113	101
9		0.08	2000	112	101
10		0.07	1986	115	102
End	627.4				

Test Phase 2, Period 4, Interval 1
Barometric Pressure 23.13 in. Hg
Sample Zone Pressure 22.99 in. Hg
Ambient Temperature 123°F

Sample Location: OG
Water Catch

Silica Gel	gross	225.6 g	
	tare	200.0 g	
	net		25.6 mL
Impinger	#1		43.7 mL
	#2		18.4 mL
	#3		3.5 mL
Cyclone			92.4 mL
Total Water			183.6 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	629.1	0.08	1870	78	72
2		0.08	1838	98	83
3		0.09	1993	105	90
4		0.09	2000	109	95
5		0.09	2000	110	96
6		0.08	2000	109	97
7		0.09	2010	109	97
8		0.09	2010	108	96
9		0.08	2005	108	96
10		0.08	1990	108	97
End	762.0				

Test Phase 2, Period 4, Interval 2
Barometric Pressure 23.07 in. Hg
Sample Zone Pressure 22.93 in. Hg
Ambient Temperature 123°F

Sample Location: OG
Water Catch

Silica Gel	gross	253.9 g	
	tare	200.0 g	
	net		53.9 mL
Impinger	#1		42.0 mL
	#2		7.9 mL
	#3		- mL
Cyclone			85.1 mL
Total Water			188.9 mL

Traverse Point	Dry Gas Meter (ft^3)	Pitot ΔP (in. W.G.)	Sample Zone Temp (°F)	Meter Temperature Inlet (°F)	Meter Temperature Outlet (°F)
1	765.5	0.08	1900	88	70
2		0.08	1872	98	84
3		0.09	2000	107	94
4		0.09	1995	112	100
5		0.08	2010	114	103
6		0.09	2000	114	102
7		0.09	2010	113	101
8		0.09	2005	110	98
9		0.08	1920	110	97
10		0.08	1924	108	96
End	889.8				

APPENDIX E

COMBUSTION EFFICIENCY AND OFFGAS COMPOSITION DATA

Carbon dioxide and carbon monoxide readings were tabulated following each incinerator feed cycle at the point where combustion efficiency (CE) was expected to be lowest. These values were then used to calculate instantaneous combustion efficiencies from

$$CE = \frac{CO_2}{CO_2 + CO} \times 100 \ .$$

The data and calculated combustion efficiencies are tabulated on the following pages and summarized in Table E-1.

The carbon dioxide and carbon monoxide data were averaged for each sample interval for use in the offgas and sample flow calculations (Appendix F) and are tabulated on the gas composition data sheets following the combustion efficiency summary.

Test (P-P-I)*	Feed Cycle	CO_2 (%)	CO (ppm)	Combustion Eff. (%)
1-1-1	1	4.8	36	99.93
	2	4.9	32	99.93
	3	5.2	34	99.93
	4	5.3	27	99.95
	5	5.4	30	99.94
	6	4.7	37	99.92
	7	4.9	32	99.93
	8	5.4	29	99.95
	9	4.8	30	99.94
	10	4.9	27	99.94
	11	5.1	30	99.94
	12	4.6	21	99.95
	Avg	5.0	30	
1-1-2	1	3.7	29	99.92
	2	4.5	21	99.95
	3	5.2	39	99.93
	4	5.0	20	99.96
	5	4.8	25	99.95
	6	4.5	30	99.93
	7	5.0	32	99.94
	8	5.1	25	99.95
	9	4.5	18	99.96
	10	4.7	20	99.96
	11	4.6	14	99.97
	12	4.8	30	99.94
	Avg	4.7	25	
1-2-1	1	5.4	34	99.94
	2	4.8	30	99.94
	3	4.7	36	99.92
	4	5.1	27	99.95
	5	4.5	39	99.91
	6	4.8	32	99.93
	7	5.6	37	99.93
	8	5.1	21	99.96
	9	5.3	18	99.97
	10	5.6	30	99.95
	11	4.8	39	99.92
	12	4.6	36	99.92
	13	4.7	34	99.93
	14	5.2	32	99.94
	15	4.8	39	99.92
	16	4.6	27	99.94
	17	5.2	23	99.96
	18	5.2	20	99.96
	Avg	4.9	31	

*P-P-I = Phase-Period-Interval.

Test (P-P-I)*	Feed Cycle	CO_2 (%)	CO (ppm)	Combustion Eff. (%)
1-2-2	1	3.8	25	99.93
	2	4.8	18	99.96
	3	4.9	36	99.93
	4	4.9	32	99.93
	5	5.2	34	99.93
	6	4.8	41	99.91
	7	5.0	32	99.94
	8	4.9	29	99.94
	9	4.2	36	99.91
	10	4.6	43	99.91
	11	4.8	30	99.94
	12	4.9	39	99.92
	13	4.9	25	99.95
	14	5.0	34	99.93
	15	5.2	45	99.91
	16	4.9	32	99.93
	17	4.8	34	99.93
	Avg	4.8	33	
2-1-1	1	4.8	32	99.93
	2	5.4	37	99.93
	3	5.5	34	99.94
	4	5.3	25	99.95
	5	4.9	21	99.96
	6	5.5	27	99.95
	7	5.5	32	99.94
	8	4.8	18	99.96
	9	5.2	30	99.94
	10	5.1	37	99.93
	11	4.8	30	99.94
	12	5.5	36	99.93
	13	5.3	27	99.95
	14	5.4	23	99.96
	15	5.4	27	99.95
	16	5.0	29	99.94
	Avg	5.1	29	

Test (P-P-I)*	Feed Cycle	CO_2 (%)	CO (ppm)	Combustion Eff. (%)
2-1-2	1	4.8	29	99.94
	2	5.4	21	99.96
	3	5.8	23	99.96
	4	5.9	27	99.95
	5	5.3	18	99.97
	6	5.0	21	99.96
	7	4.7	25	99.95
	8	5.4	29	99.95
	9	5.1	23	99.95
	10	5.6	23	99.96
	11	5.0	29	99.94
	12	4.9	27	99.94
	13	5.7	18	99.97
	14	5.8	20	99.97
	15	5.5	23	99.96
	16	5.6	21	99.96
	17	5.4	18	99.97
	18	5.8	23	99.96
	Avg	5.4	23	
2-2-1	1	5.4	32	99.94
	2	5.0	25	99.95
	3	4.8	18	99.96
	4	5.1	20	99.96
	5	5.5	21	99.96
	6	5.3	23	99.96
	7	4.8	29	99.94
	8	5.0	34	99.93
	9	5.5	25	99.95
	10	5.5	23	99.96
	11	5.3	18	99.97
	12	5.0	20	99.96
	13	5.4	21	99.96
	Avg	5.2	24	

Test (P-P-I)*	Feed Cycle	CO_2 (%)	CO (ppm)	Combustion Eff. (%)
2-2-2	1	5.4	21	99.96
	2	5.0	27	99.95
	3	5.3	23	99.96
	4	5.2	34	99.93
	5	5.5	36	99.93
	6	5.5	39	99.93
	7	4.8	30	99.94
	8	5.0	25	99.95
	9	5.1	32	99.94
	10	5.3	34	99.94
	11	5.5	37	99.93
	12	4.8	25	99.95
	13	5.4	29	99.95
	14	5.0	21	99.96
	Avg	5.2	30	
2-3-1	1	5.2	32	99.94
	2	4.8	29	99.94
	3	4.7	34	99.93
	4	5.1	36	99.93
	5	5.2	27	99.95
	6	4.8	23	99.95
	7	5.2	21	99.96
	8	5.5	27	99.95
	9	4.9	18	99.96
	10	4.7	20	99.96
	11	5.0	29	99.94
	12	5.1	25	99.95
	13	4.9	30	99.94
	14	4.7	29	99.94
	15	5.3	21	99.96
	16	5.4	18	99.97
	17	4.8	16	99.97
	18	4.8	18	99.96
	Avg	5.0	25	

Test (P-P-I)*	Feed Cycle	CO_2 (%)	CO (ppm)	Combustion Eff. (%)
2-3-2	1	4.7	32	99.93
	2	4.9	30	99.94
	3	5.1	21	99.96
	4	5.3	27	99.95
	5	5.0	29	99.94
	6	4.7	34	99.93
	7	4.5	32	99.93
	8	5.1	37	99.93
	9	4.9	32	99.93
	10	5.3	29	99.95
	11	5.0	23	99.95
	12	4.5	30	99.93
	13	4.8	27	99.94
	14	4.9	32	99.93
	15	5.2	37	99.93
	16	5.1	29	99.94
	17	4.7	25	99.95
	18	4.5	20	99.96
	19	4.9	27	99.94
	Avg	4.8	29	
2-4-1	1	5.9	37	99.94
	2	5.5	32	99.94
	3	5.4	21	99.96
	4	5.4	16	99.97
	5	5.8	25	99.96
	6	5.3	30	99.94
	7	5.5	21	99.96
	8	5.6	34	99.94
	9	5.5	37	99.93
	10	5.7	43	99.92
	11	5.4	29	99.95
	12	5.5	21	99.96
	13	5.6	27	99.95
	Avg	5.6	29	

Test (P-P-I)*	Feed Cycle	CO_2 (%)	CO (ppm)	Combustion Eff. (%)
2-4-2	1	5.0	27	99.95
	2	5.3	21	99.96
	3	4.9	23	99.95
	4	6.0	75	99.86**
	5	5.5	39	99.93
	6	4.8	29	99.94
	7	4.9	59	99.88**
	8	5.6	32	99.94
	9	4.8	21	99.95
	10	5.5	29	99.95
	11	5.5	18	99.97
	12	5.3	16	99.97
	Avg	5.3	28	

**Occurred during fast shutdown transient.

Gas Compositions (Dry Basis)

Test Phase 1, Period 1, Interval 1

	Location	
Component	HZ	OG
O_2 (%)	6.0	11.0
CO (ppm)	---*	30
CO_2 (%)	---	5
N_2 (%)	---	84.0

Test Phase 1, Period 1, Interval 2

	Location	
Component	HZ	OG
O_2 (%)	6.0	9.0
CO (ppm)	---	25
CO_2 (%)	---	4.7
N_2 (%)	---	86.3

Test Phase 1, Period 2, Interval 1

	Location	
Component	HZ	OG
O_2 (%)	6.0	8.0
CO (ppm)	---	31
CO_2 (%)	---	4.9
N_2 (%)	---	87.1

*Indicates not measured.

Gas Compositions (Dry Basis) (cont)

Test Phase 1, Period 2, Interval 2

	Location	
Component	HZ	OG
O_2 (%)	6.0	8.0
CO (ppm)	---	33
CO_2 (%)	---	4.8
N_2	---	87.2

Test Phase 2, Period 1, Interval 1

	Location	
Component	HZ	OG
O_2 (%)	4.0	8.0
CO (ppm)	---	29
CO_2	---	5.1
N_2	---	86.9

Test Phase 2, Period 1, Interval 2

	Location	
Component	HZ	OG
O_2 (%)	5.0	7.8
CO (ppm)	---	23
CO_2 (%)	---	5.4
N_2 (%)	---	86.8

Test Phase 2, Period 2, Interval 1

	Location	
Component	HZ	OG
O_2 (%)	5.0	7.5
CO (ppm)	---	24
CO_2 (%)	---	5.2
N_2 (%)	---	87.3

Gas Compositions (Dry Basis) (cont)

Test Phase 2, Period 2, Interval 2

	Location	
Component	HZ	OG
O_2 (%)	6.0	7.0
CO (ppm)	---	30
CO_2 (%)	---	5.2
N_2 (%)	---	87.8

Test Phase 2, Period 3, Interval 1

	Location	
Component	HZ	OG
O_2 (%)	5.0	6.5
CO (ppm)	---	25
CO_2 (%)	---	5.0
N_2 (%)	---	88.5

Test Phase 2, Period 3, Interval 2

	Location	
Component	HZ	OG
O_2 (%)	5.0	6.0
CO (ppm)	---	28
CO_2 (%)	---	4.8
N_2 (%)	---	89.2

Test Phase 2, Period 4, Interval 1

	Location	
Component	HZ	OG
O_2 (%)	1.0	7.5
CO (ppm)	---	29
CO_2 (%)	---	5.6
N_2 (%)	---	86.9

Test Phase 2, Period 4, Interval 2

	Location	
Component	HZ	OG
O_2 (%)	0.5	8.0
CO (ppm)	---	28
CO_2 (%)	---	5.3
N_2 (%)	---	86.7

TABLE E-1. COMBUSTION EFFICIENCY CALCULATIONS SUMMARY

Sample Interval	Min. (%)	Avg (%)
1-1-1*	99.92	99.939
1-1-2	99.92	99.946
1-2-1	99.91	99.938
1-2-2	99.91	99.931
2-1-1	99.93	99.944
2-1-2	99.94	99.956
2-2-1	99.93	99.954
2-2-2	99.93	99.943
2-3-1	99.93	99.950
2-3-2	99.93	99.941
2-4-1	99.92	99.948
2-4-2	99.86**	99.939

*Test Phase-Test Period-Test Interval.
**Occurred during a fast shutdown transient.

APPENDIX F

SAMPLE AND FLOW CALCULATIONS SUMMARY

The field data (Appendix D) and the gas composition data (Appendix E) were used in the calculation of offgas flows and sample volumes as described below.

Variable definitions:

- SA = sample zone flow area (432 in.^{2} for HZ, 314 in.^{2} for OG).
- V_m = sample volume at meter conditions.
- V_s = sample volume at standard conditions on dry basis.
- V_w = volume of water vapor in sample at standard conditions.
- T_i = meter inlet temperature.
- T_o = meter outlet temperature.
- T_m = average meter temperature.
- $V_{w\%}$ = per cent moisture in offgas.
- M_d = mole fraction dry offgas.
- MW_g = molecular weight of offgas.
- EA = excess air (%).
- T_s = temperature of offgas at sample zone.
- S = average (square root of velocity head $\times$ T_s).
- H_v = velocity head (pressure drop on pitot).
- u = offgas velocity in sample zone at sample zone conditions.
- Q = volumetric flow of offgas at standard conditions.
- $\overline{[\ ]}$ = concentration in per cent, i.e., $\overline{[CO]}$ = per cent CO.
- V_m = dry gas meter reading at end of sample interval minus dry gas meter reading at beginning of sample interval.

$$T_i(\text{average}) = \left[\sum_{j=1,n} T_i(j) \right] / n \text{ where n is the number of traverse points .}$$

$$T_o(\text{average}) = \left[\sum_{j=1,n} T_o(j) \right] / n \ .$$

$$T_m = [T_i(\text{average}) + T_o(\text{average})]/2 \ .$$

$$V_s = 17.7\, V_m \times \frac{\text{pressure at meter}}{T_m + 460} \ .$$

$$V_w = 0.0474 \times \text{mL water trapped in sample train} \ .$$

$$V_{w\%} = 100\, V_w/(V_s + V_w) \ .$$

$$M_d = (100 - V_{w\%})/100 \ .$$

$$MW_g = (0.44[CO_2] + 0.28[CO] + 0.32[O_2] + 0.28[N_2])M_d + 18(1 - M_d) \ .$$

$$EA = 100([O_2] - [CO]/2)/(0.264[N_2] - [CO]/2) \ .$$

$$T_s(\text{average}) = \left[\sum_{j=1,n} T_s(j)\right] / n \ .$$

$$S = \left(\sum_{j=1,n} (H_v(j) \times [T_s(j) + 460])^{0.5}\right) / n \ .$$

$$u = 4350 \ S \ [1/(MW_g \times \text{sample zone pressure})]^{0.5} \ .$$

$$\underline{Q} = 0.123 \ u \ SA \ M_d \times \text{sample zone pressure}/(T_s \text{ average} + 460) \ .$$

In the above calculations, n = 12 for hot-zone (HZ) samples and 10 for hot crossover duct (OG) samples. Results of the calculations are summarized in the following tabulation.

Test (P-P-I)*	Sample Zone**	T_m (°F)	V_m (ft^3)	V_s (ft^3)	EA (%)	u (ft/min)	$\underline{Q}$ (ft^3/min)
1-1-1	HZ	64	104.3	81.4	179	454	250
1-1-2	HZ	90	100.3	74.7	34	534	274
1-2-1	HZ	90	107.3	79.8	21	589	298
1-2-2	HZ	84	111.4	83.8	41	544	275
2-1-1	HZ	87	87.7	65.4	48	555	296
2-1-2	HZ	87	105.4	78.8	30	545	290
2-2-1	HZ	87	120.5	91.3	57	530	286
2-2-2	HZ	89	130.8	97.4	46	571	298
2-3-1	HZ	93	120.4	88.9	32	580	290
2-3-2	HZ	83	80.9	60.9	25	564	283
2-4-1	HZ	94	116.2	86.0	31	547	281
2-4-2	HZ	85	91.4	68.6	65	594	317
1-1-1	OG	93	123.5	91.5	98	2140	835
1-1-2	OG	92	99.3	73.5	65	1889	743
1-2-1	OG	83	21.3	16.0	53	2206	785
1-2-2	OG	101	126.2	92.0	53	2274	847
2-1-1	OG	102	82.9	60.3	54	2387	830
2-1-2	OG	107	113.4	81.7	52	2325	839
2-2-1	OG	104	130.1	94.4	48	2507	862
2-2-2	OG	102	103.5	75.4	43	2520	857
2-3-1	OG	105	116.9	84.5	39	2461	802
2-3-2	OG	105	148.7	107.4	34	2443	806
2-4-1	OG	98	132.9	97.7	49	2452	822
2-4-2	OG	101	124.3	90.6	54	2456	817

* P-P-I = Phase-Period-Interval.

**Sample zones were HZ = hot zone (primary chamber offgas) and OG = hot crossover duct (secondary chamber offgas).

APPENDIX G

REPORT ON SAMPLE ANALYSIS FROM SOUTHWEST RESEARCH INSTITUTE

This report has been reproduced as received from SWRI.

CLASSIFICATION

SOUTHWEST RESEARCH INSTITUTE

POST OFFICE DRAWER 28510 · 6220 CULEBRA ROAD · SAN ANTONIO, TEXAS 78284 · (512) 684-5111

DIVISION OF CHEMISTRY
AND CHEMICAL ENGINEERING

January 12, 1982

Dr. Larry Stretz
University of California
Los Alamos Laboratory
Box 990; Lab SP-2
Los Alamos, New Mexico 97545

Subject: SwRI Project 01-6761-023

Dear Dr. Stretz:

Attached please find the report on the methods and results of the GC/MS analyses of sample extracts sent to us. If you have any questions or require further information, please call me.

Very truly yours,

Carter Nulton, Ph.D.
Manager, Mass Spectrometry

APPROVED:

Donald E. Johnson, Director
Department of Environmental Sciences

CN:bz/L7
Attachments

CLASSIFICATION

Methods

Sample extract number 1 was broken on receipt at SwRI.

All extracts were ca. 30 mL, except number 7 which was ca. 23 mL. Extracts were reduced in volume to 0.5 mL under a gentle stream of chromatographic grade nitrogen. The internal standards d_8-naphthalene, d_{10}-anthracene and d_{12}-chrysene were added to the concentrated extracts prior to injection; the final concentrations of the internal standards were 51, 44 and 39 ng/µL, respectively.

The instrument used for these analyses was a Finnigan 3623 quadrapole mass spectrometer equipped with an INCOS data system and a Tracor 560 GC. The fused silica capillary column is threaded through a heated conduit into the mass spectrometer ion source.

The following compounds were searched for by extracted ion current profiling in each total ion current (TIC) run:

chlorophenols - mono through pentachloro
chlorobenzenes - di through hexachloro
chlorobenzenes - di through hexachloro
chlorodibenzodioxins - mono through octachloro
chlorodibenzofurans - mono through octachloro

The following instrument operating conditions were used for the TIC runs:

GC

Column	15 m X 0.25 mm fused silica capillary
Phase	SE-54; 1.0 µ film
Carrier gas	He at 40 cm/sec
Temperature program	65°|1 min|10/min|300°
Injector temperature	280°
Splitless injection	

MS

Electron energy	70 ev
Scan rate	1 sec/scan

Extracts were reanalyzed with the mass spectrometer in the selected ion monitor (SIM) mode; the target compounds were pentachlorophenol (m/e 266, 268) tetrachlorodibenzodioxin (m/e 320, 322) and tetrachloro-

dibenzofuran (m/e 304, 306). Other instrument operating conditions were identifical to those listed for the TIC runs.

Selected target compound detection limits for both the TIC and SIM analyses are given in Table 1.

Results

Target compounds and their concentrations found in the TIC runs are listed in Table 2.

In Table 3 tentatively identified major peaks are given along with estimated concentrations (relative to nearest internal standard). The TIC chromatograms are attached.

Except for the pentachlorophenol found in sample extract number 113 none of the SIM target compounds were detected in the SIM runs.

TABLE 1
SELECTED TARGET COMPOUND DETECTION LIMITS

	Total µg in Sample[a]
TIC	
2-Chlorophenol	1.3
2,4-dichlorophenol	3
trichlorophenol	6
hexachlorobenzene	6
2-chlorodibenzo-p-dioxin	5
2,8-dichlorodibenzofuran	5
2,7-dichlorodibenzo-p-dioxin	6
pentachlorophenol	10
1,2,4-trichlorodibenzo-p-dioxin	8
1,2,3,4-tetrachlorodibenzo-p-dioxin	8
octachlorodibenzo-p-dioxin	20
octachlorodibenzofuran	20
MID	
pentachlorophenol	5
tetrachlorodibenzo-p-dioxin	.5
tetrachlorodibenzofuran	.5

a - Based on a final sample volume of 0.5 mLs and a 2 µL injection volume.

TABLE 2
TARGET COMPOUNDS DETECTED IN TIC RUNS

	Total µg in Sample
Sample 110	
1,3-dichlorobenzene	tr
1,4-dichlorobenzene	tr
1,2-dichlorobenzene	tr
2-chlorophenol	tr
1,2,4-trichlorobenzene	0.9
1,2,3-trichlorobenzene	tr
1,2,3,5 or 1,2,4,5-tetrachlorobenzene	tr
1,2,3,4-tetrachlorobenzene	tr
pentachlorobenzene	tr
Sample 111	
1,3-dichlorobenzene	tr
1,4-dichlorobenzene	tr
1,2-dichlorobenzene	tr
1,2,4-trichlorobenzene	0.9
1,2,3-trichlorobenzene	tr
1,2,3,5 or 1,2,4,5-tetrachlorobenzene	tr
1,2,3,4-tetrachlorobenzene	tr
pentachlorobenzene	tr
Sample 112	
1,4-dichlorobenzene	tr
1,2,4-trichlorobenzene	tr
Sample 113	
pentachlorophenol	61,000
tetrachlorophenol	16,000

tr = trace, below detection limits

TABLE 3
TENTATIVE IDENTIFICATIONS AND RELATIVE CONCENTRATIONS
OF MAJOR PEAKS IN TIC RUNS

Scan Number		Total ug in Sample
Sample 2, 5, 7, 30, 27 had no major peaks		
Sample 6		
524	naphthalene[a]	>2.5
628	naphthalene 2-methyl	<2.5
705	1,1'-biphenyl	<2.5
779	acenaphthylene	>2.5
893	9H-fluorene	>2.5
106	phenanthrene[b]	<2.5
1163	4H-cyclopenta[def]phenanthrene	<2.5
1268	fluoranthene	~2.5
1307	pyrene	~2.5
1482	benzo[ghi]fluoroanthene or isomer	<2.5
1519	benzo[ghi]fluoroanthene or isomer	<2.5
1538	bis(2-ethylhexyl)phthalate	~2.5
1856	benzofluoroanthene or isomer	<2.5
Sample 10		
526	naphthalene[a]	>2.5
629	naphthalene, 2-methyl	<2.5
707	1,1'-biphenyl	<2.5
782	acenaphthylene	>2.5
837	dibenzofuran	<2.5
895	9H-fluorene	<2.5
1062	phenanthrene[b]	>2.5
1164	4H-cyclopenta[def]phenanthrene	>2.5
1269	fluoranthene	>2.5
1309	pyrene	<2.5
1446	hexanedioicacid, dioctylester	<2.5
1483	benzo[ghi]fluoranthene or isomer	<2.5
1520	benzo[ghi]fluoranthene or isomer	~2.5
1539	bis(2-ethylhexyl)phthalate	<2.5
1767	benzofluoranthene or isomer	<2.5
1859	benzofluoranthene or isomer	<2.5
Sample 11		
366	benzyl alcohol	~2.5
523	naphthalene[a]	>2.5
778	acenaphthylene	~2.5
1060	phenanthrene[b]	~2.5

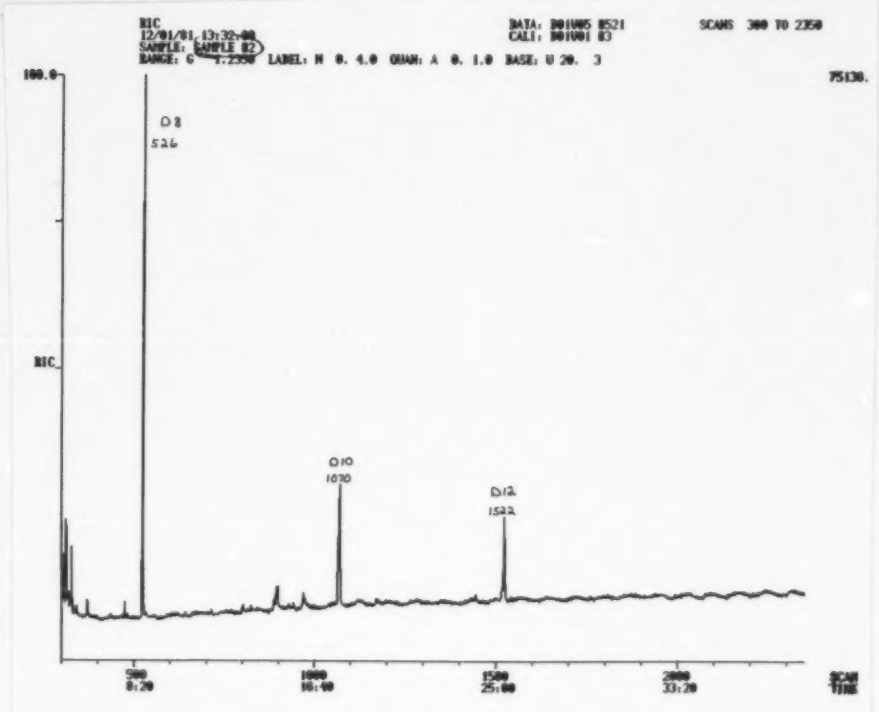
Scan Number		Total ug in Sample
Sample 22		
1266	fluoranthene	~2.5
1305	pyrene	~2.5
1447	hexandioic acid, bis(2-ethylhexyl)ester	>2.5
Sample 23		
361	benzyl alcohol	<2.5
1446	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
Sample 26		
381	benzene, 1-propynyl or 1H-indene	<2.5
525	naphthalene[a]	>2.5
781	acenaphthylene	>2.5
1061	phenanthrene[b]	>2.5
1268	fluoranthene	~2.5
1306	pyrene	2.5
1446	hexanediocacid, bis(2-ethyl)ester	~2.5
Sample 34		
369	benzyl alcohol	<2.5
781	acenaphthylene	<2.5
1435	butylbenzylphthalate	<2.5
1449	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
1543	bis(2-ethylhexyl)phthalate	>2.5
Sample 38		
364	benzyl alcohol	<2.5
524	naphthalene[a]	>2.5
782	acenaphthylene	>2.5
1270	fluoranthene	~2.5
1308	pyrene	~2.5
1450	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
Sample 42		
314	methylphenol	<2.5
432	naphthalene[a]	>2.5
690	acenaphthylene	~2.5
966	phenanthrene[b]	<2.5
1167	fluoranthene	~2.5
1204	pyrene	~2.5
1345	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
1434	bis(2-ethylhexyl)phthalate	>2.5

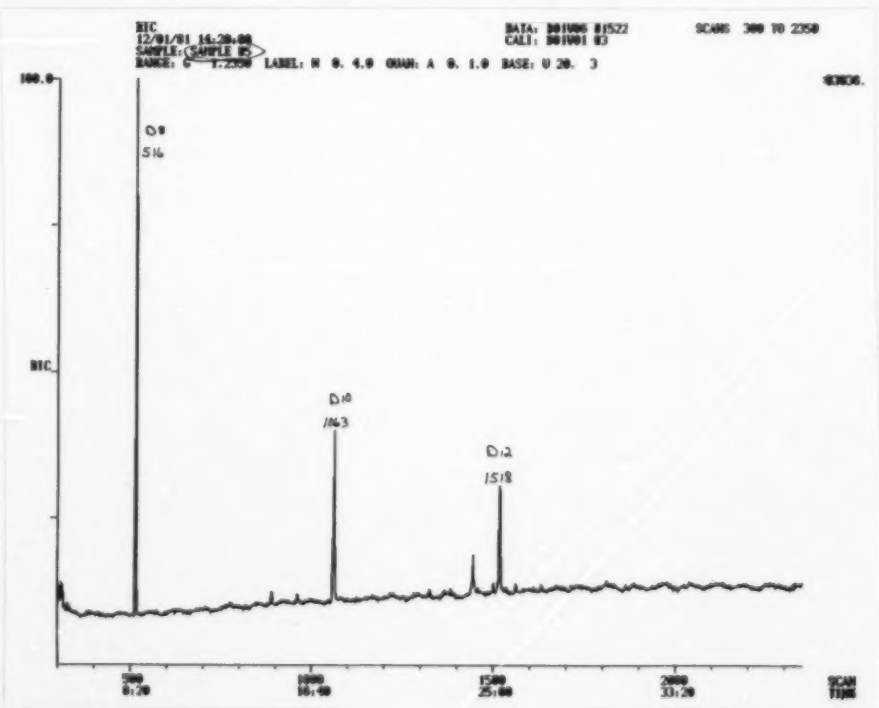
Scan Number		Total ug in Sample
Sample 43		
333	benzyl alcohol	<2.5
1397	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
Sample 46		
339	naphthalene[a]	>2.5
564	acenaphthylene	~2.5
673	9H-fluorene	<2.5
832	phenanthrene[b]	>2.5
1037	fluoranthene	>2.5
1074	pyrene	>2.5
1221	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
1310	bis(2-ethylhexyl)phthalate	~2.5
Sample 47		
923	aliphatic hydrocarbon	<2.5
1401	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
Sample 50		
325	benzylalcohol	<2.5
484	naphthalene[a]	>2.5
603	methylnaphthalene	<2.5
664	1,1-biphenyl	<2.5
734	acenaphthylene	~2.5
848	9H-fluorene	<2.5
1011	phenanthrene[b]	>2.5
1216	fluoranthene	>2.5
1254	pyrene	>2.5
1400	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
1429	benzo[g,h,i]fluoranthene	<2.5
1464	benzofluoroanthene or isomer	~2.5
1737	benzofluoranthene or isomer	~2.5
Sample 51		
329	benzylalcohol	<2.5
1398	hexanediocacid, bis(2-ethylhexyl)ester	>2.5
Sample 106		
325	1-hexanol, 2-ethyl	>2.5
333	benzylalcohol	>2.5
522	1,2-benzisothiazole	<2.5
1488	bis(2-ethylhexyl)phthalate	>2.5

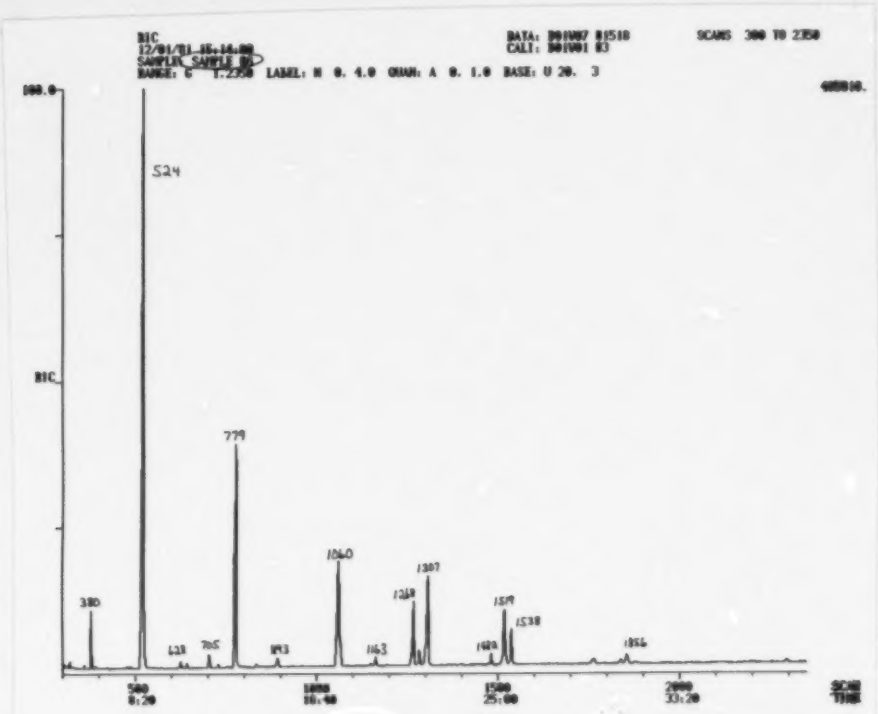
Scan Number		Total ug in Sample
Sample 110		
299	trimethyl benzene	<2.5
323	1-hexanol, 2-ethyl	~2.5
485	naphthalene[a]	>2.5
523	1,2-benzisothiazole	<1.5
	scans 700-1500 envelope of incompletely resolved hydrocarbons	~2.5 each
805	trimethyl naphthalene	~2.5
1493	bis(2-ethylhexyl)phthalate	>2.5
Sample 111		
303	trimethyl benzene	<2.5
327	1-hexanol, 2-ethyl	~2.5
335	benzyl alcohol	~2.5
525	1,2-benzisothiazole	<2.5
	scans 700-1200 envelope of imcompletely resolved hydrocarbons	<2.5 each
712	dimethylnaphthalene	<2.5
806	trimethylnaphthalene	<2.5
1497	bis(2-ethylhexyl)phthalate	>2.5
Sample 112		
326	1-hexanol, 2-ethyl	<2.5
333	benzyl alcohol	<2.5
1500	bis(2-ethylhexyl)phthalate	>2.5
	branched and/or cyclic hydrocarbons	>2.5
	aliphatic hydrocarbon series	>2.5
1000	pentachlorophenol	(see Table 2)
1504	bis(2-ethylhexyl)phthalate	>2.5

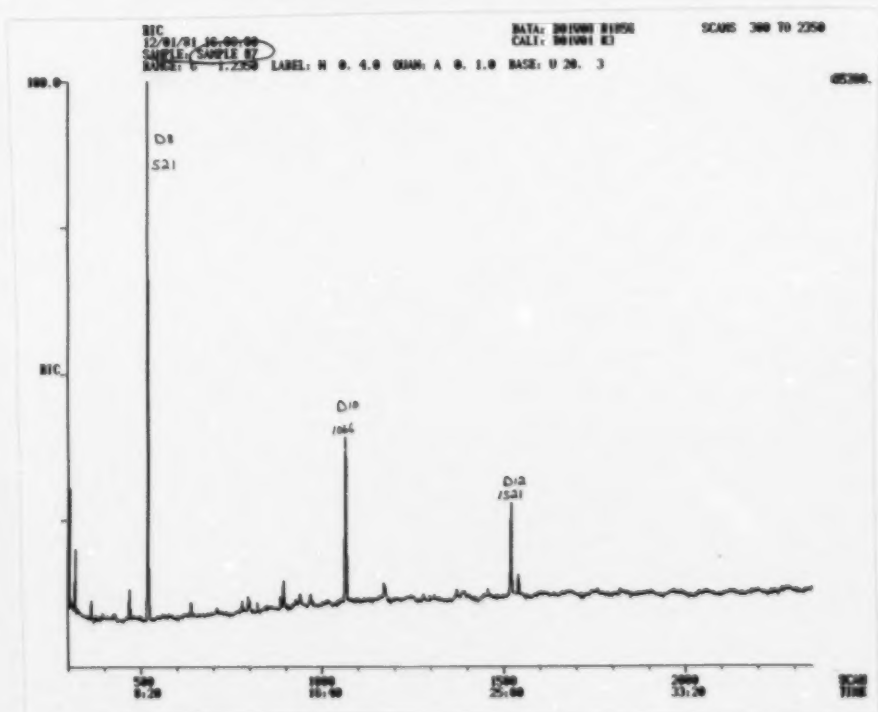
a - co-elutes with d_8-naphthalene

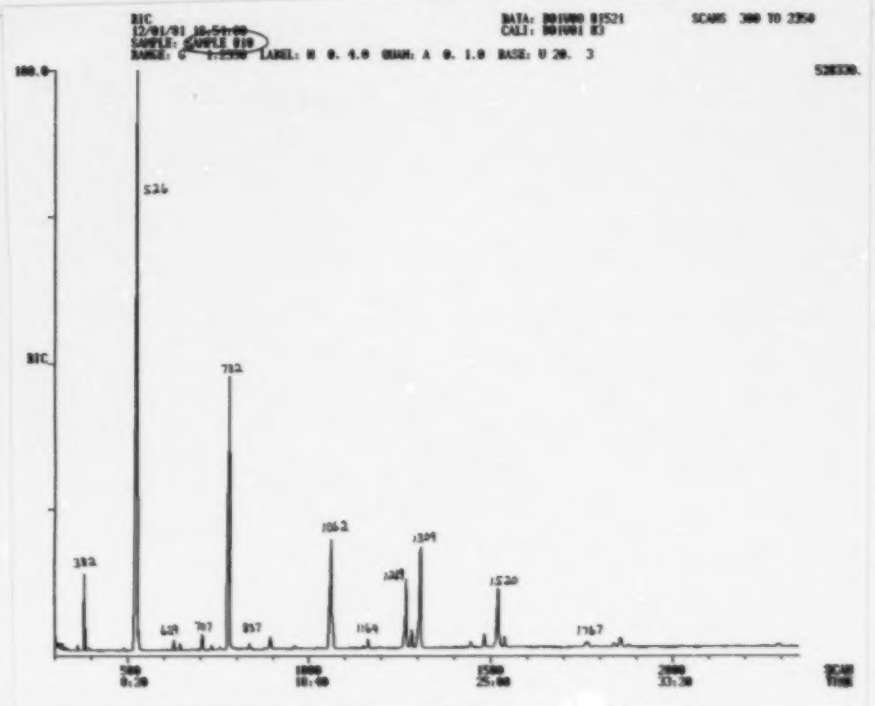
b - co-elutes with d_{10}-anthracene

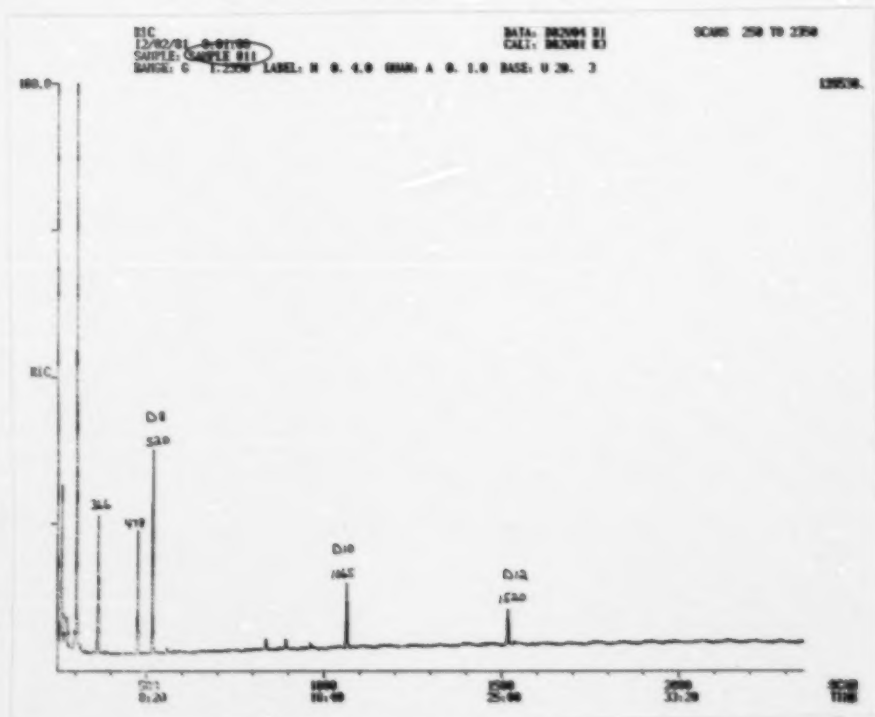

RIC
12/01/01 13:32:00
SAMPLE: SAMPLE #2
DATA: 001005 #521
CALI: 001001 #3
SCANS 300 TO 2350
RANGE: C 1:2350 LABEL: M 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3
100.0
75130.
RIC
D8
526
D10
1070
D12
1522
500
8:20
1000
16:40
1500
25:00
2000
33:20
SCAN
TIME


RIC
12/01/01 14:20:00
SAMPLE: SAMPLE #5
DATA: 001005 #522
CALI: 001001 #3
SCANS 300 TO 2350
RANGE: C 1:2350 LABEL: M 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3
100.0
93836.
RIC
D8
516
D10
1043
D12
1518
500
8:20
1000
16:40
1500
25:00
2000
33:20
SCAN
TIME

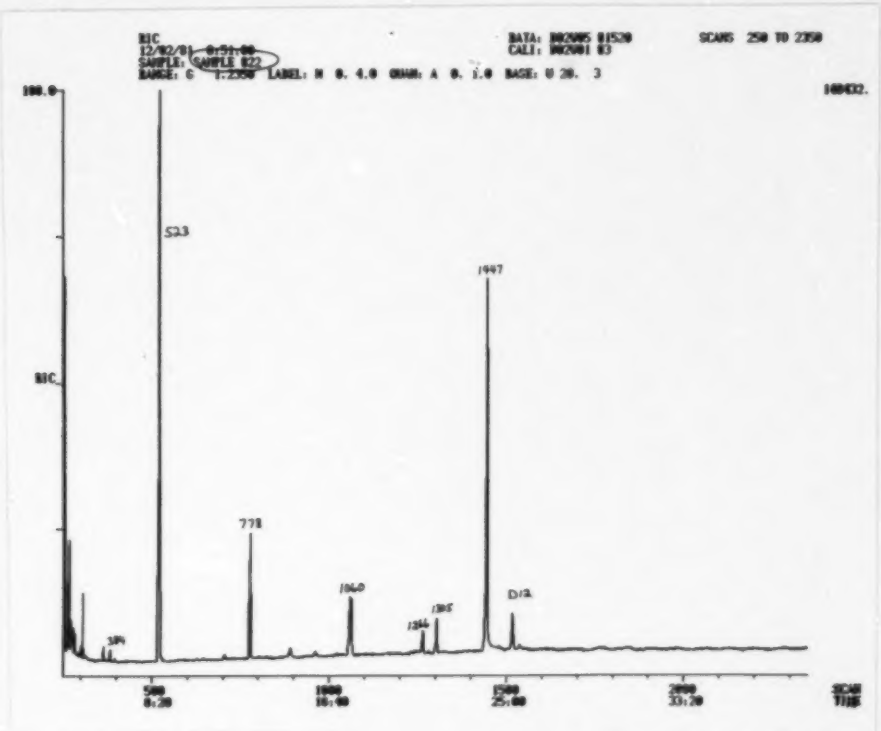

TIC
SCANS 300 TO 2350
100.0
524
TIC
779
1040
380
1307
1538
1356
500
1000
1500
2000

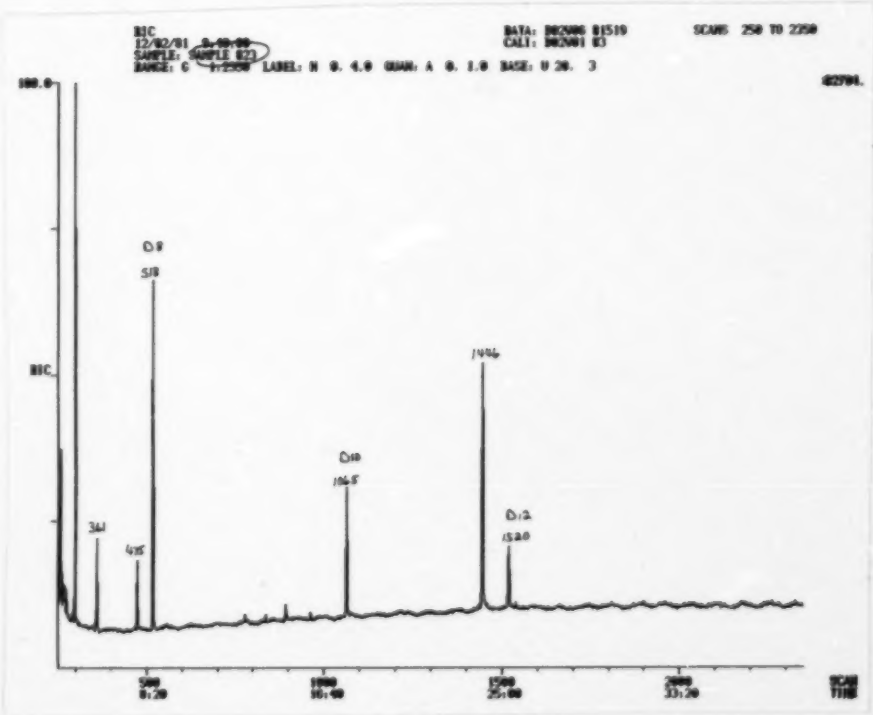

TIC
SCANS 300 TO 2350
100.0
D8
521
TIC
D10
1044
D12
1531
500
1000
1500
2000

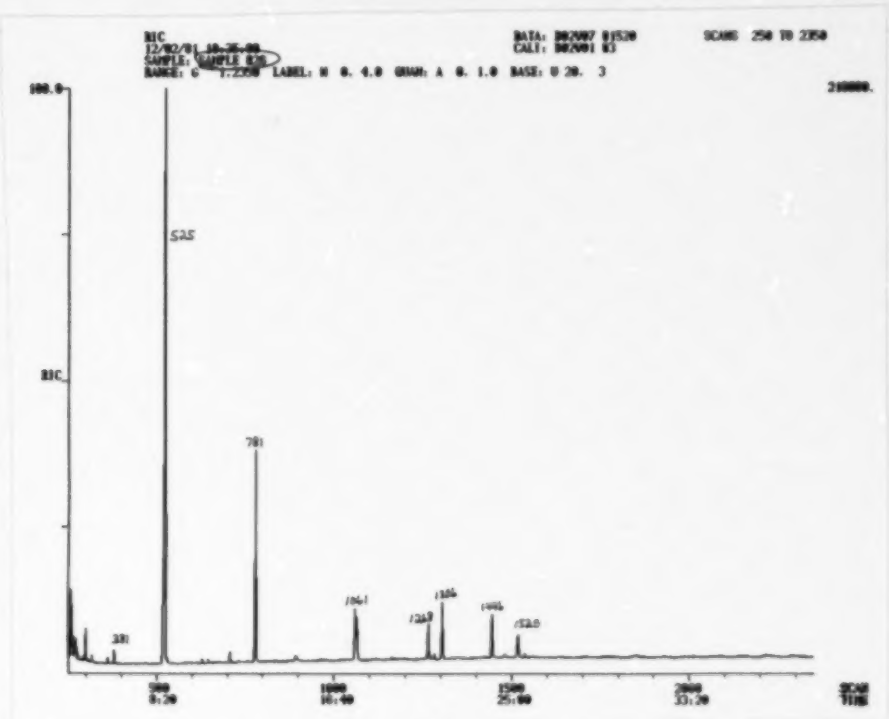


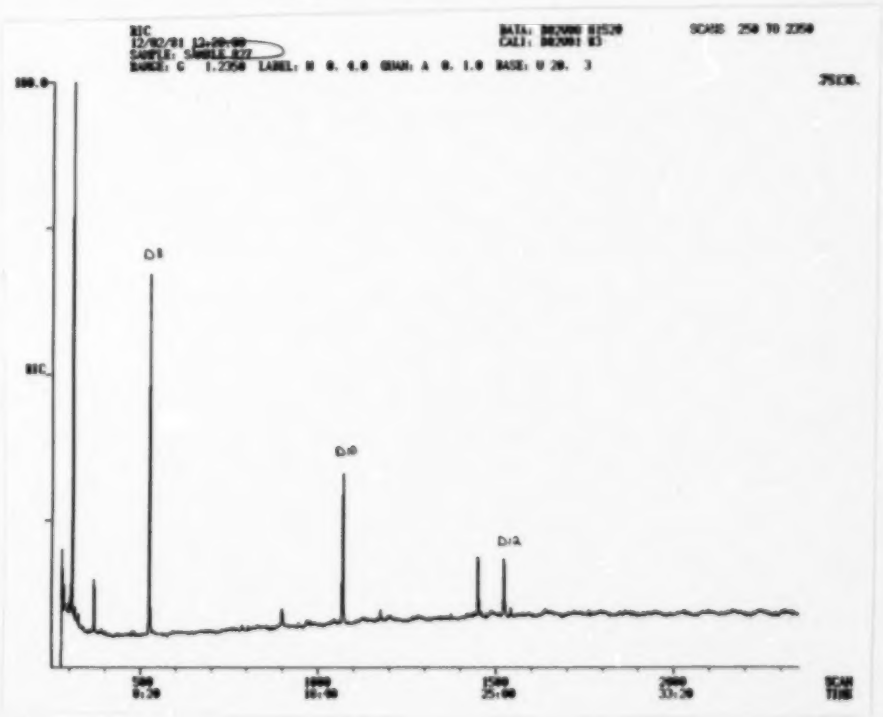


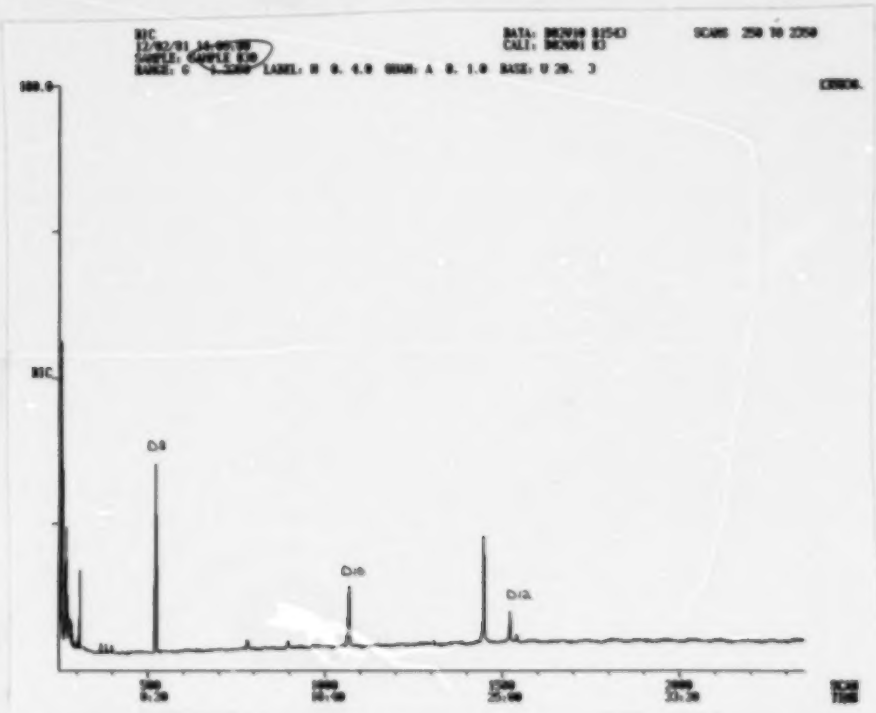
Reproduced from best available copy.

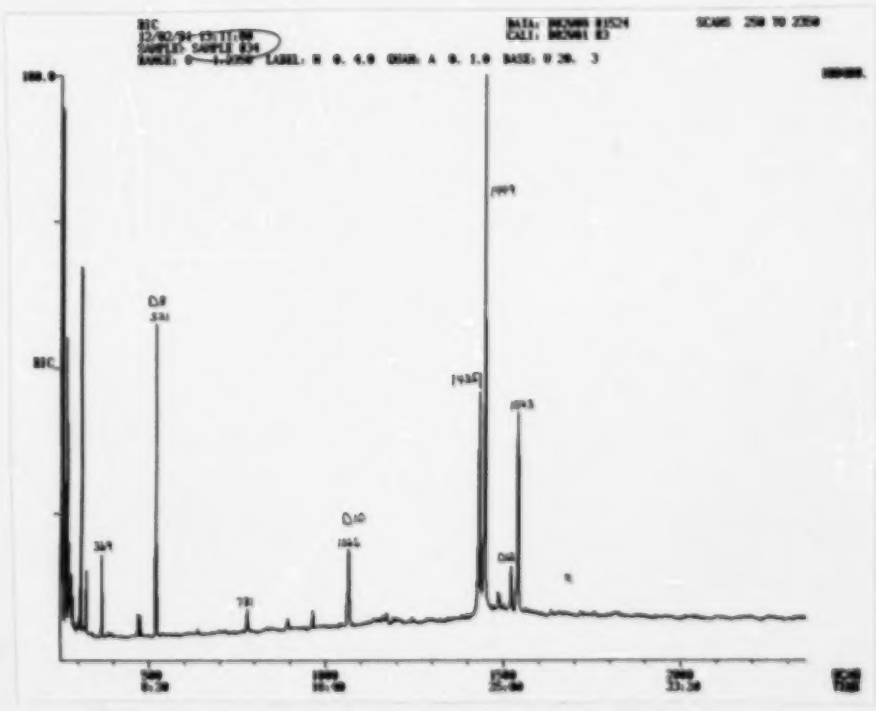

TIC
SCANS 250 TO 2250
100.0
523
1997
778
1060
1344
1395
D12
TIC

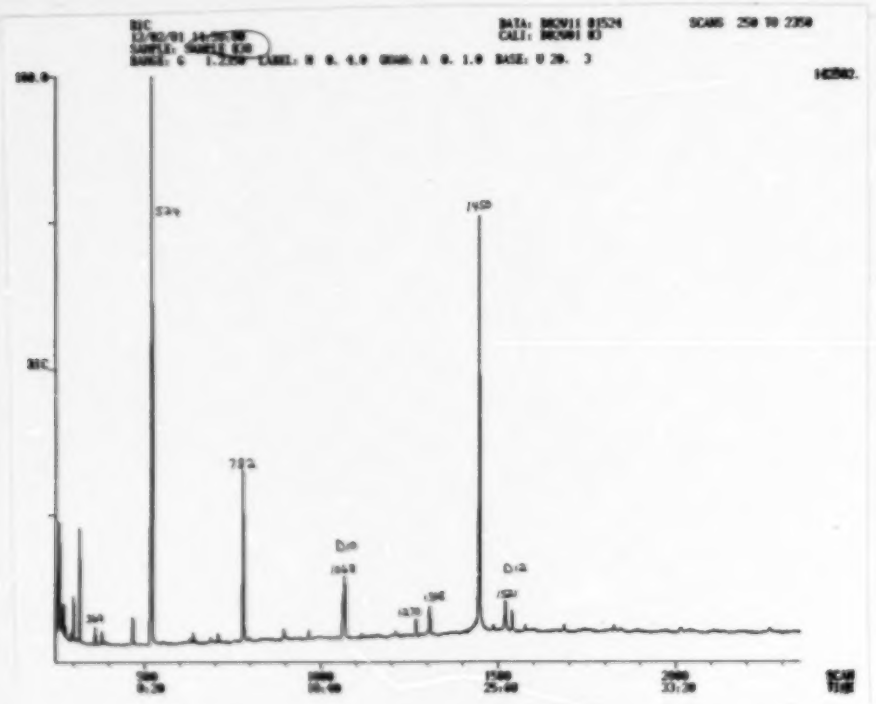

TIC
SCANS 250 TO 2250
100.0
D9
518
1996
D10
1065
341
435
D12
1520
TIC

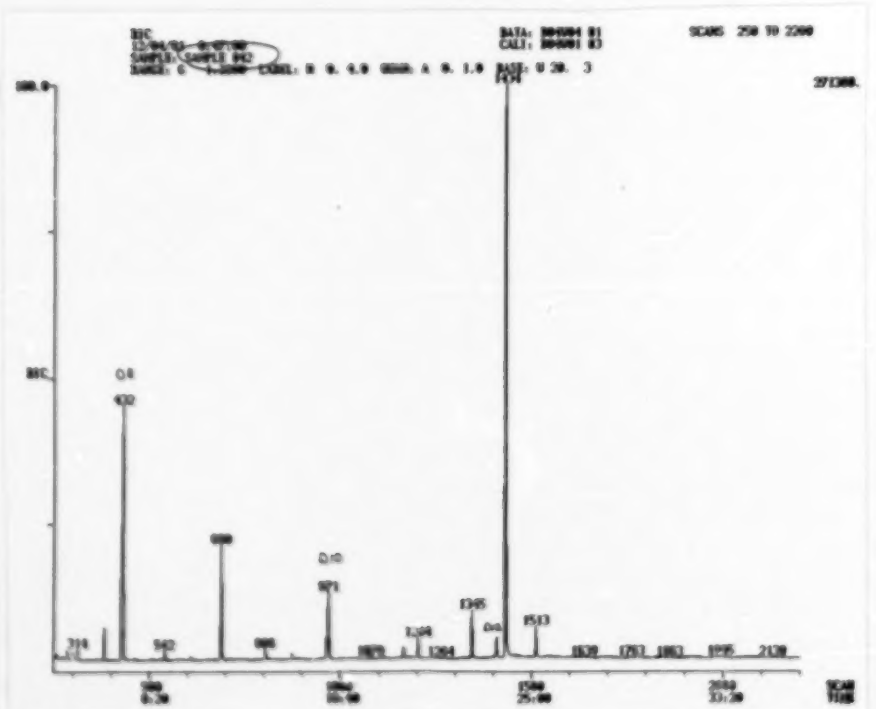

SAS
781
1061
1264
1449
1520
338

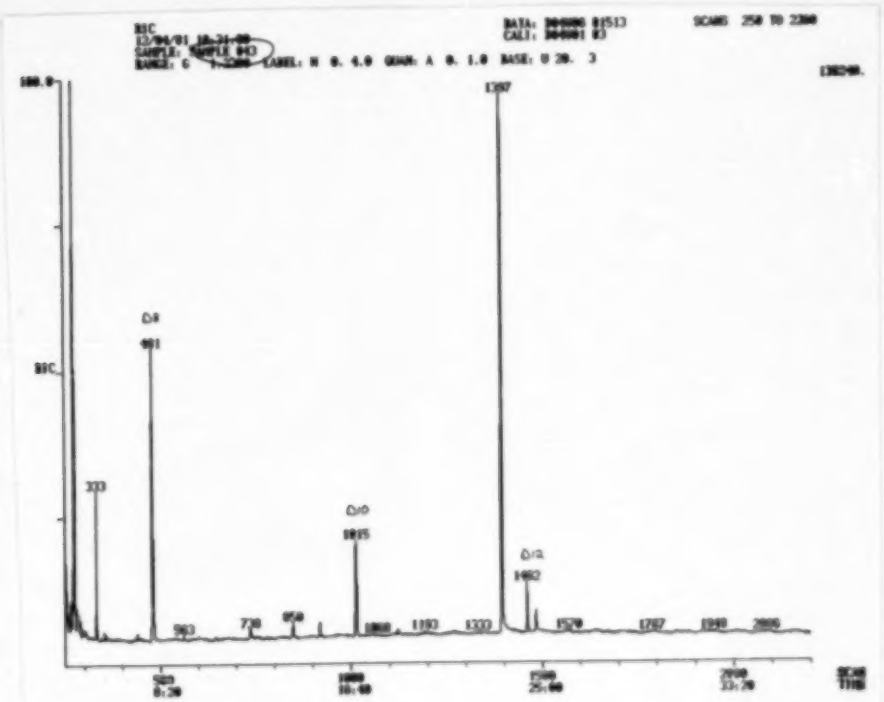

D1
D2
D3

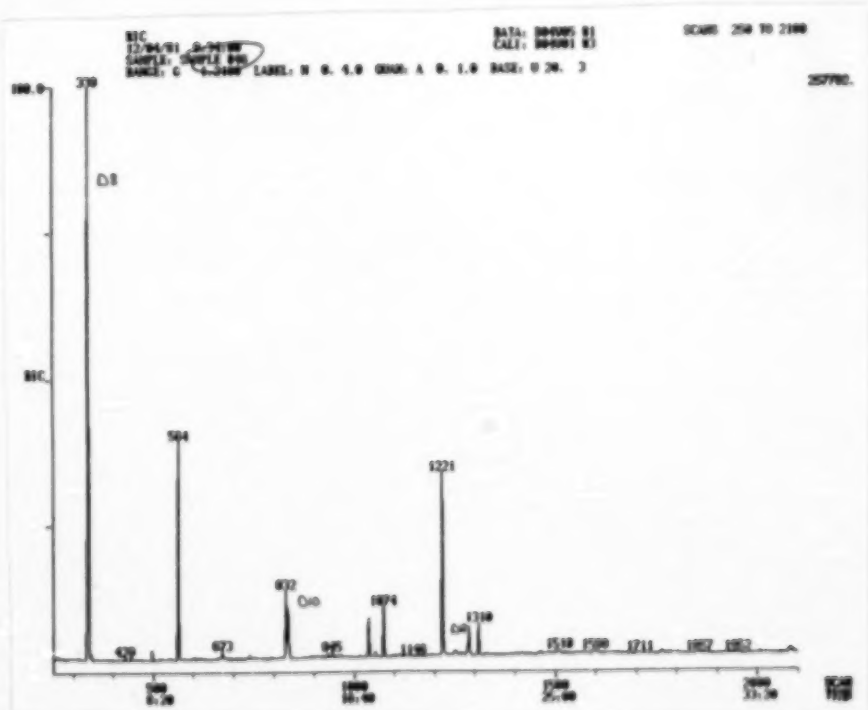

TIC
D8
D10
D12

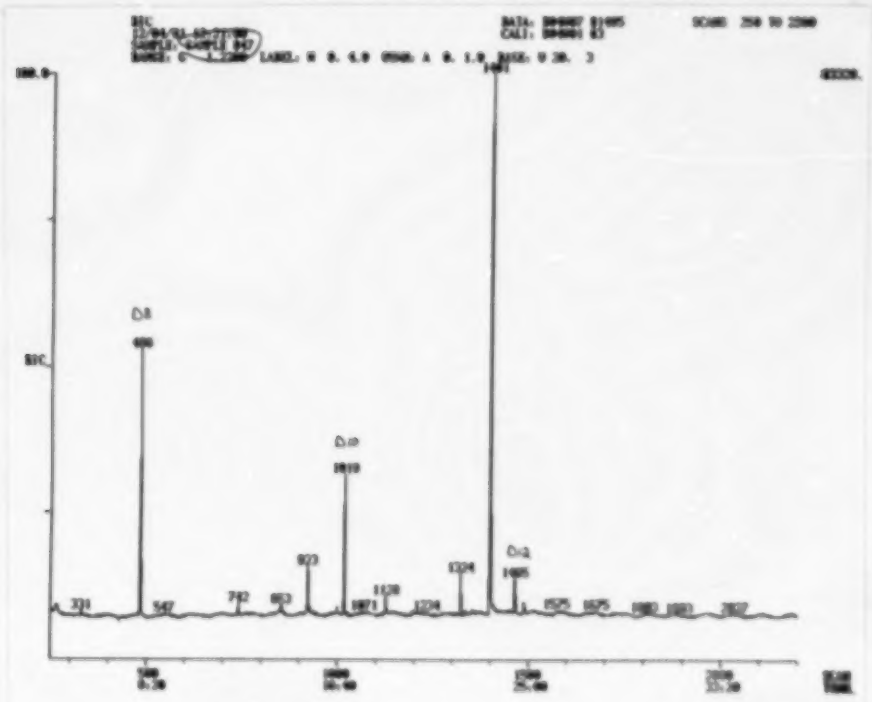

TIC
D8
D10

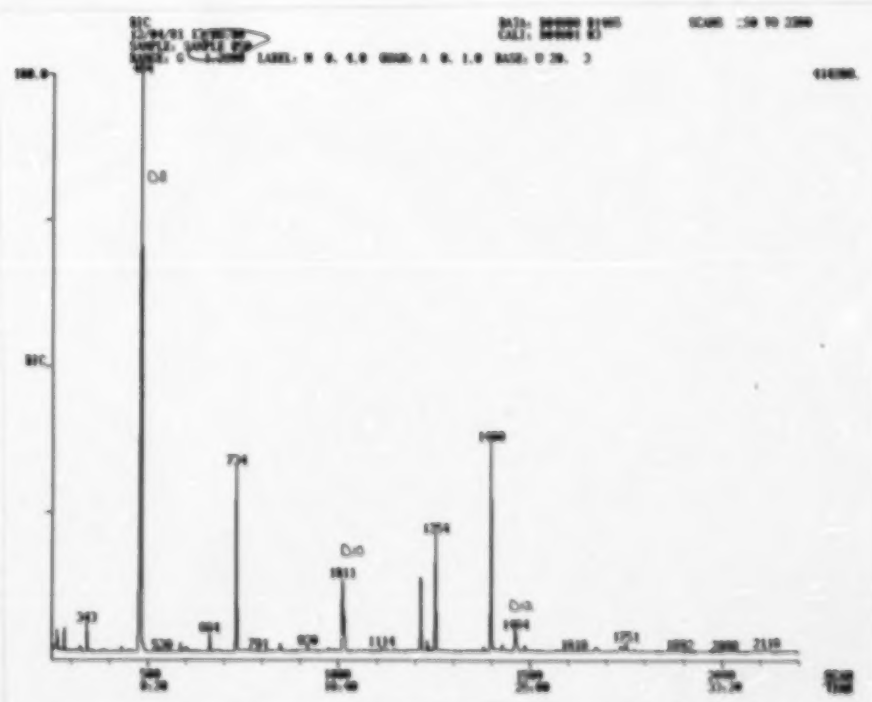


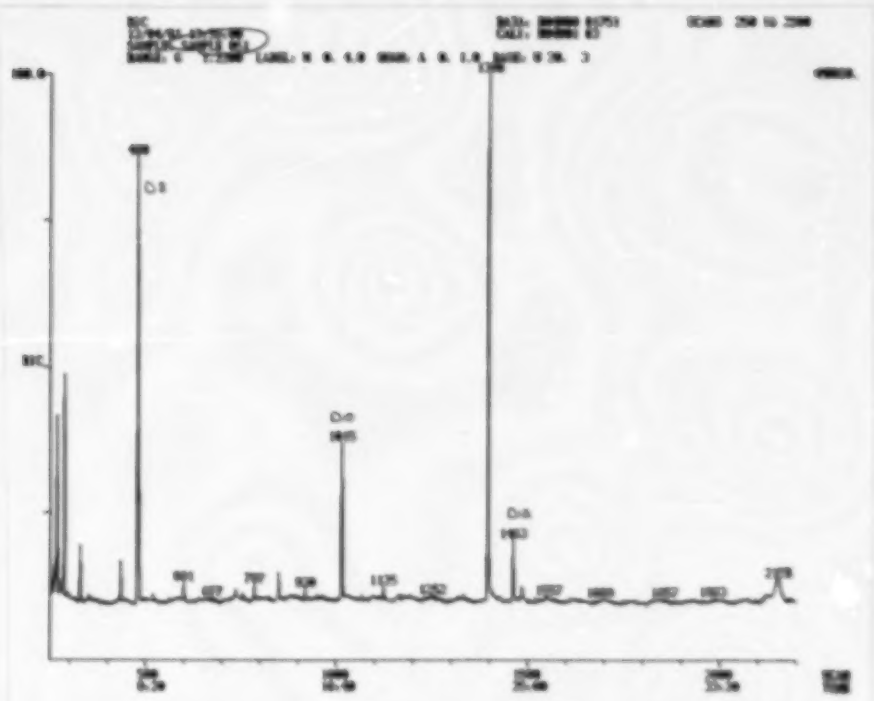


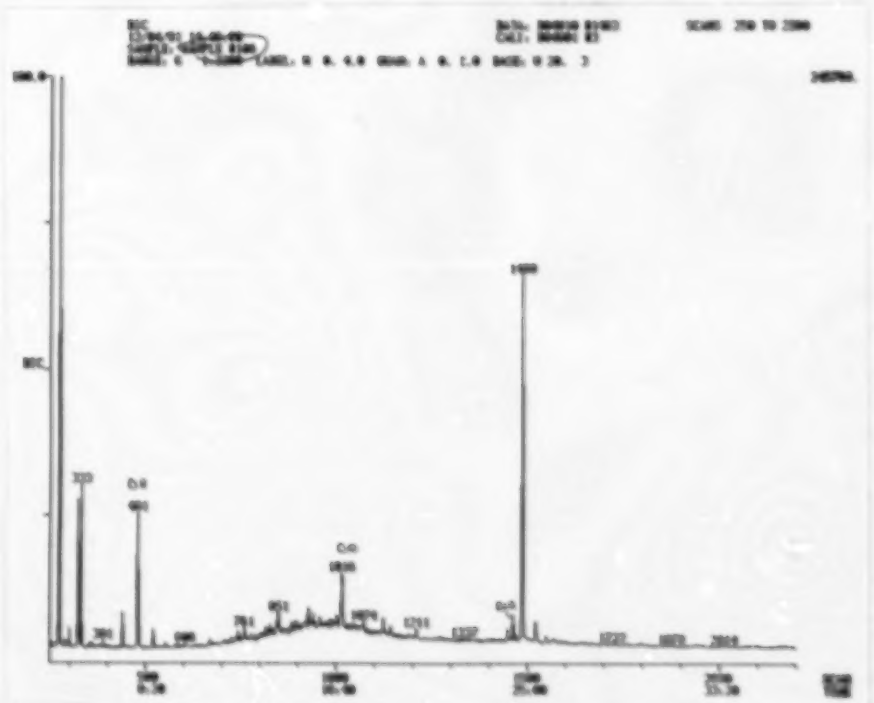


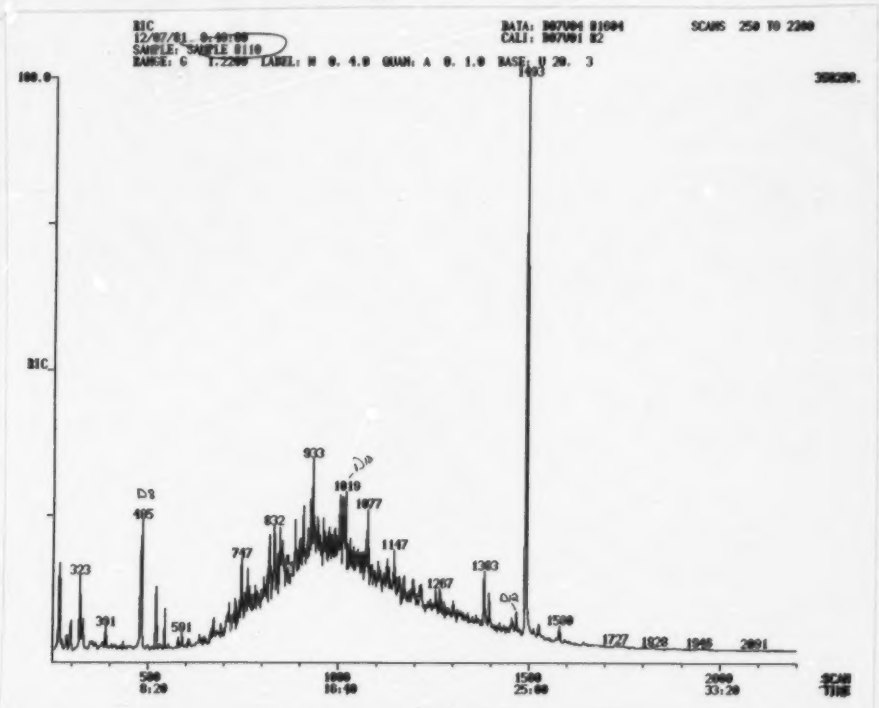


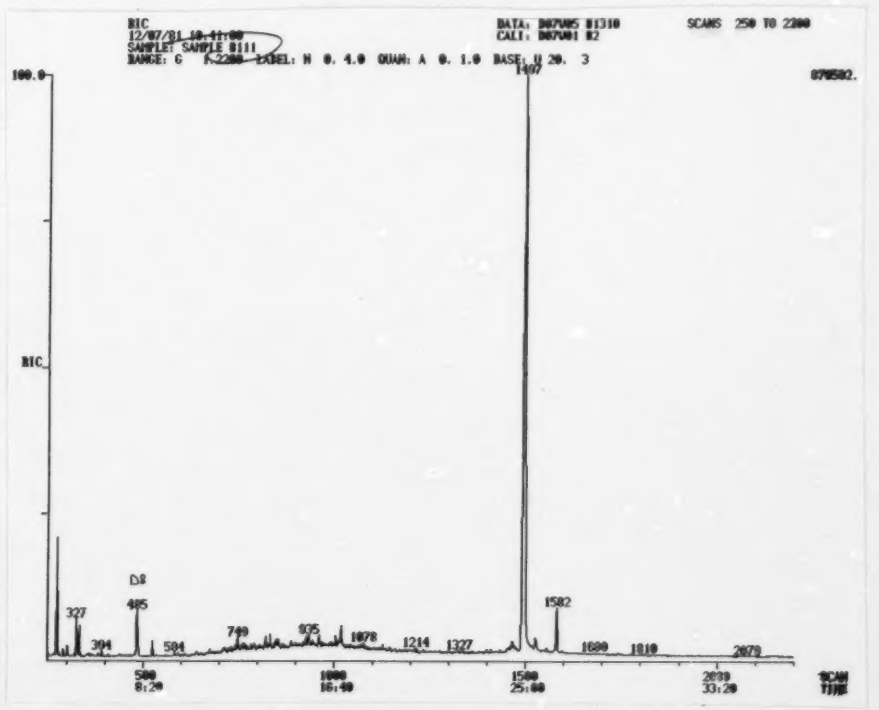


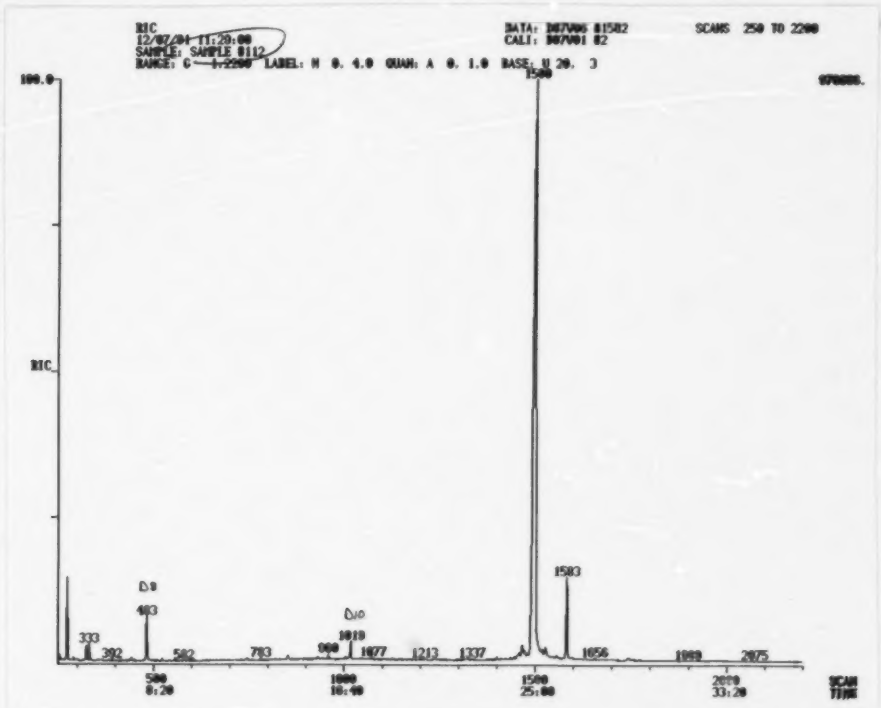


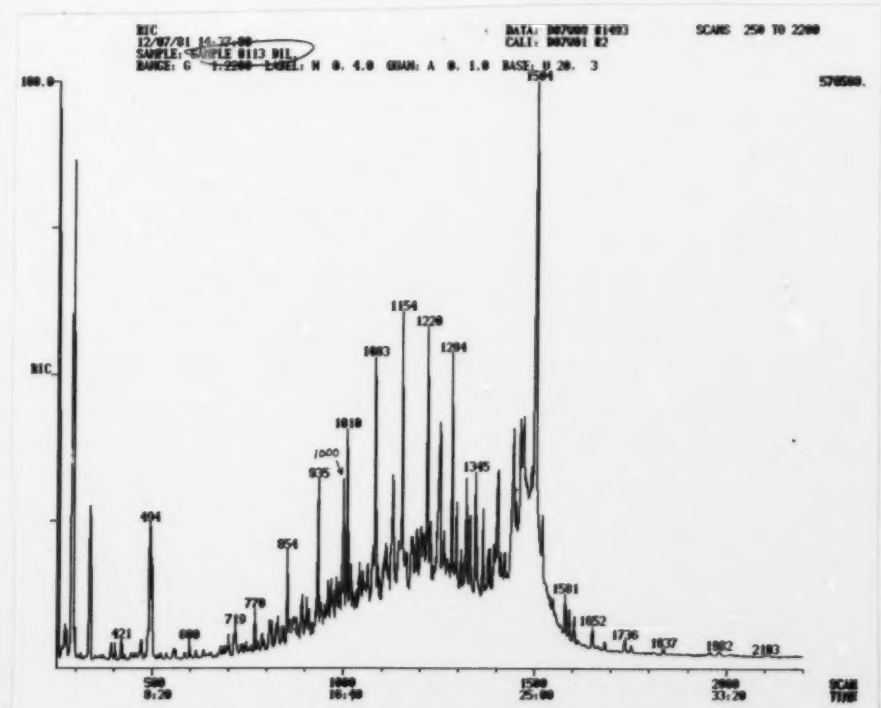





BIC
12/07/81
SAMPLE: SAMPLE 8110
DATA: D07V04 B1004
CALI: D07V01 B2
SCANS 250 TO 2200
100.0
BIC
323
391
405
501
747
822
933
1019
1077
1147
1267
1383
1493
1580
1727
1828
1948
2001
500
8:20
1000
16:40
1500
25:00
2000
33:20
SCAN TIME


BIC
12/07/81
SAMPLE: SAMPLE 8111
DATA: D07V05 B1310
CALI: D07V01 B2
SCANS 250 TO 2200
100.0
BIC
327
391
405
501
749
935
1078
1214
1327
1497
1582
1680
1810
2079
500
8:20
1000
16:40
1500
25:00
2000
33:20
SCAN TIME


RIC
12/07/01 11:20:00
SAMPLE: SAMPLE 0112
RANGE: G 1:2200 LABEL: H 0. 4.0 QUAN: A 0. 1.0 BASE: H 20. 3
DATA: 007V06 01502
CALI: 007V01 02
SCANS 250 TO 2200
100.0
970000.
RIC
1500
1503
D9
483
D10
1010
333
392
582
783
900
1077
1213
1337
1656
1888
2075
500
8:20
1000
16:40
1500
25:00
2000
33:20
SCAN
TIME


RIC
12/07/01 14:22:00
SAMPLE: SAMPLE 0113 DIL
RANGE: G 1:2200 LABEL: H 0. 4.0 QUAN: A 0. 1.0 BASE: H 20. 3
DATA: 007V00 01403
CALI: 007V01 02
SCANS 250 TO 2200
100.0
570500.
RIC
1504
1154
1220
1294
1083
1010
1000
935
1345
494
854
770
710
1581
1652
1736
1937
1982
2183
500
8:20
1000
16:40
1500
25:00
2000
33:20
SCAN
TIME